

## **Syntheses of C17-C27 fragments of 20-deoxybryostatins for assembly using Julia and metathesis reactions**

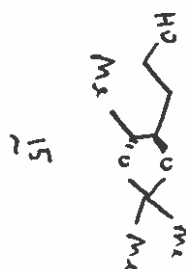
Matthew Ball, Thomas Gregson, Hiroki Omori, and Eric J. Thomas\*

### **Supplementary data**

Copies of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of some key compounds –in numerical order

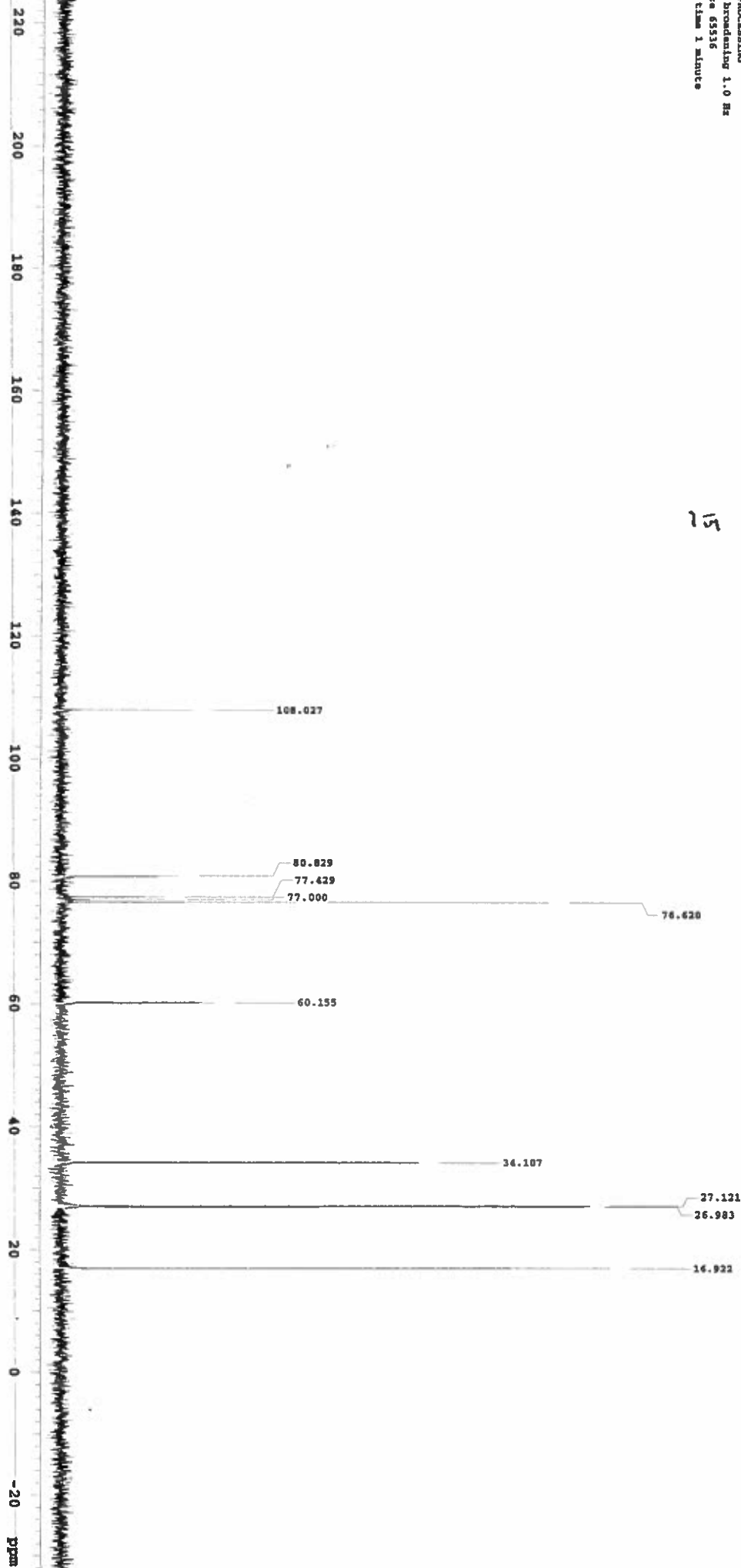
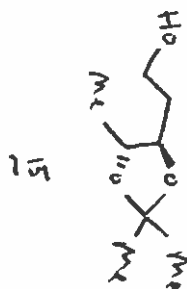
MB05/2

1  
/2/pure oil 2  
Date Oct 31  
File name /export/home/vmm2/2000/Oct31e/1301  
Solvent: cdcl3  
Ambient temperature  
Queue name par/HI  
DATE Oct 31 00  
INNOVA-300 "athos"  
PULSE SEQUENCE  
Relax, delay 3.100 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions  
OBSERVE H1, 399.6458793 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65336  
Total time 1 minutes



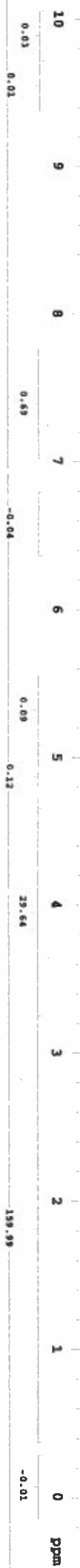
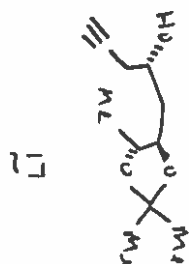
MR05/4 (28-43)  
STANDARD 1B OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #4  
File: 402  
Queue name: AD-09b  
Date: Apr 9 01  
INSTR: 300 "athos"  
PULSE SEQUENCE  
Pulse: 45.0 degrees  
Acq. time: 1.638 sec  
Width: 20000.0 Hz  
12 repetitions  
OBSERVE: C13, 75.383141 MHz  
DECOUPLE: H1, 299.843785 MHz  
Power: 40 dB  
continuously on  
NMR-16 Modulated  
DATA PROCESSING  
Line broadening: 1.0 Hz  
FT size: 65536  
Total time: 1 minute



MELOC/6/col1155-2031  
STANDARD IN OBSERVE

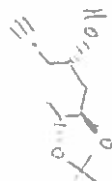
Solvent: CCl<sub>4</sub>  
Ambient temperature  
Sample #1  
File: 101  
Queue name Dec14a  
DATE Dec 14 00  
INSTR-100 "athos"  
PULSE SEQUENCE  
Relax. delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.661 sec  
Width 489.9 Hz  
16 repetitions  
OBSERVE H1, 299.845793 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 minutes



MR006/6/COU1155-203  
13C OBSERVE

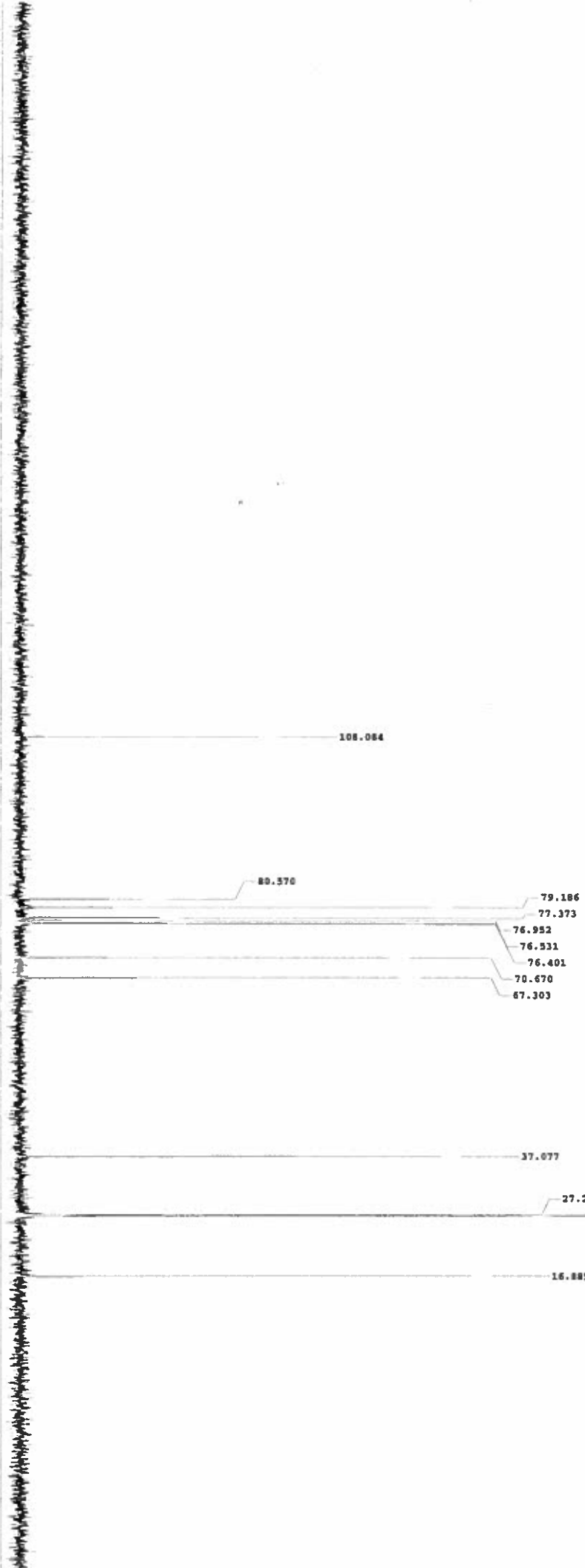
Solvent: CHCl3  
Ambient temperature  
Sample #3  
File: 301  
Queue name Dec14  
DATE Dec 14 00  
INSTR-300 "athos"

PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.618 sec  
Width 20000.0 Hz  
144 repetitions  
OBSERVE C13, 75.3963141 MHz  
DECOUPLE H1, 299.8473785 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 3 minutes



17

220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm



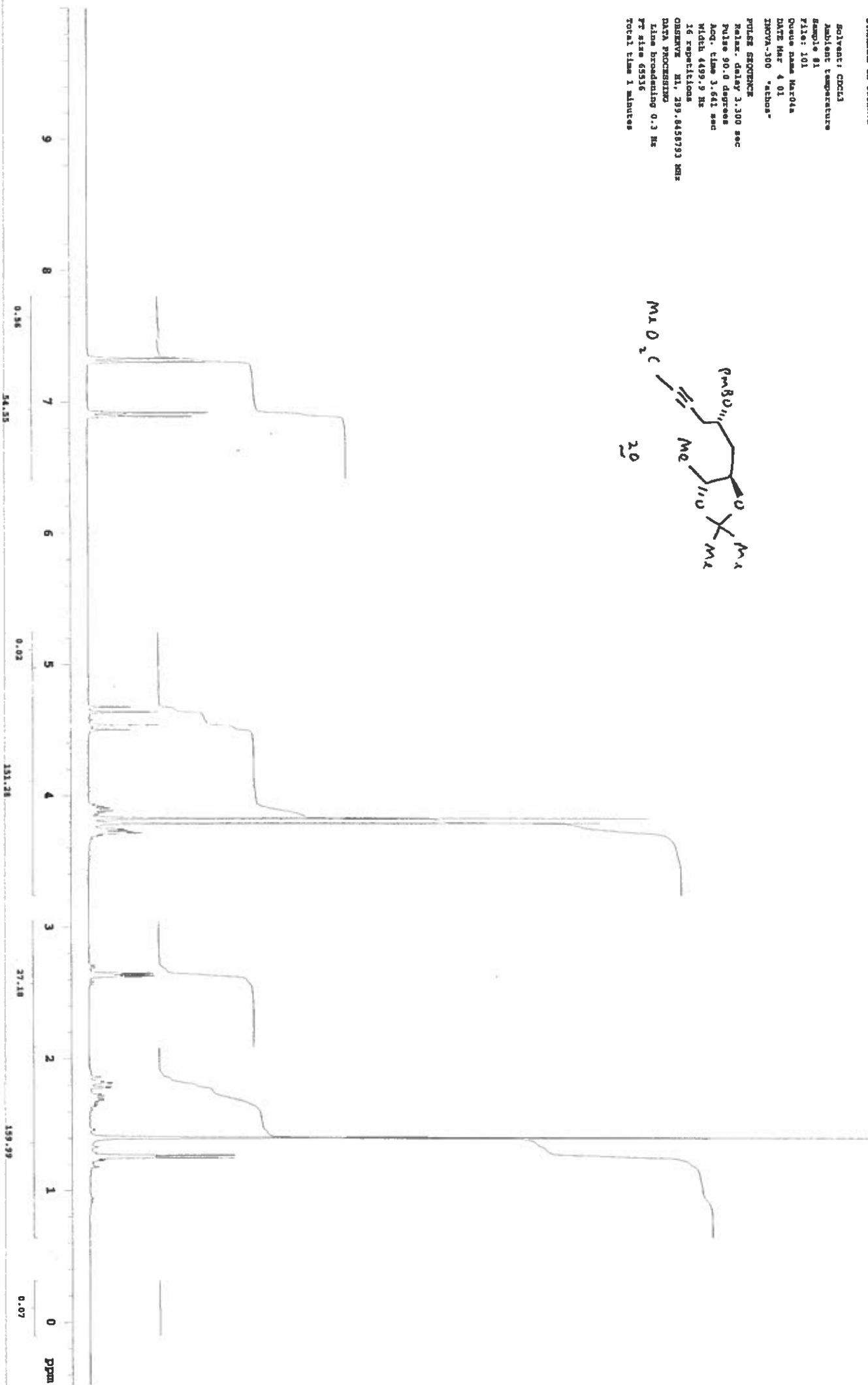
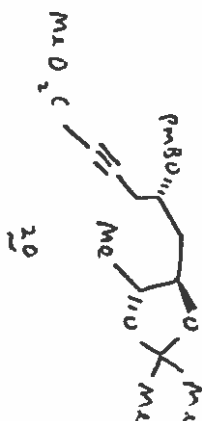
NM1030/5 (18-30)  
STANDARD IN OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #1  
File: 101  
Queue name Mar04a  
DATE Mar 4 01  
INSTR-300 "ethos"

PULSE SEQUENCE

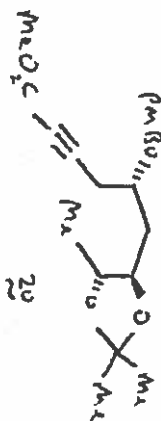
Relax. delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions

OBSERVE H1, 299.8458793 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 minutes

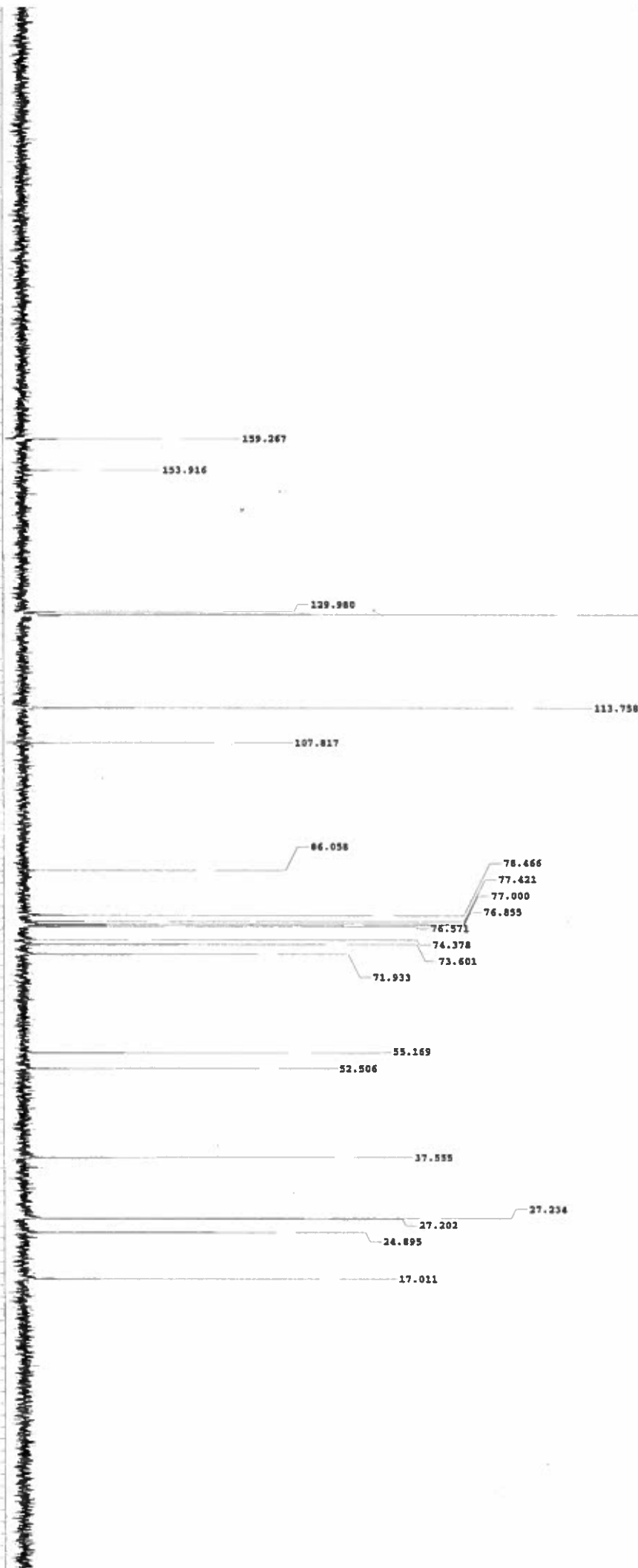


MR020/3(18-20)  
13C OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample 83  
File: 301  
Queue name Mr04a  
Date Mar 4 01  
INOVA-300 "aboa"  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.618 sec  
Width 20000.0 Hz  
112 repetitions  
OBSERVE C13, 75.3953141 MHz  
DECOUPLE H1, 299.8473785 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
PT size 65536  
Total time 3 minutes



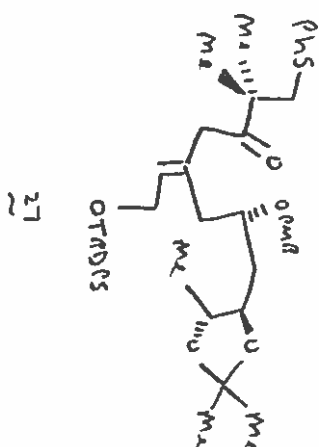
220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm



MR010/1 (6-10)  
STANDARD IR OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #4  
File: 401  
Queue name May01a  
DATE May 1 01  
INOVA-300 "alpha"

PULSE SEQUENCE  
Relax. delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions  
OBSERVE: H1, 299.8458793 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 minutes

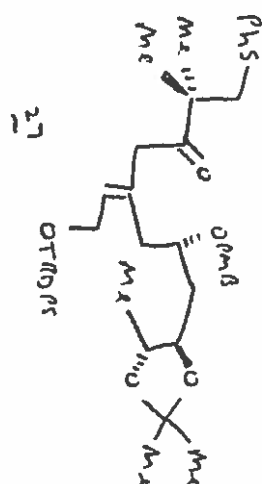
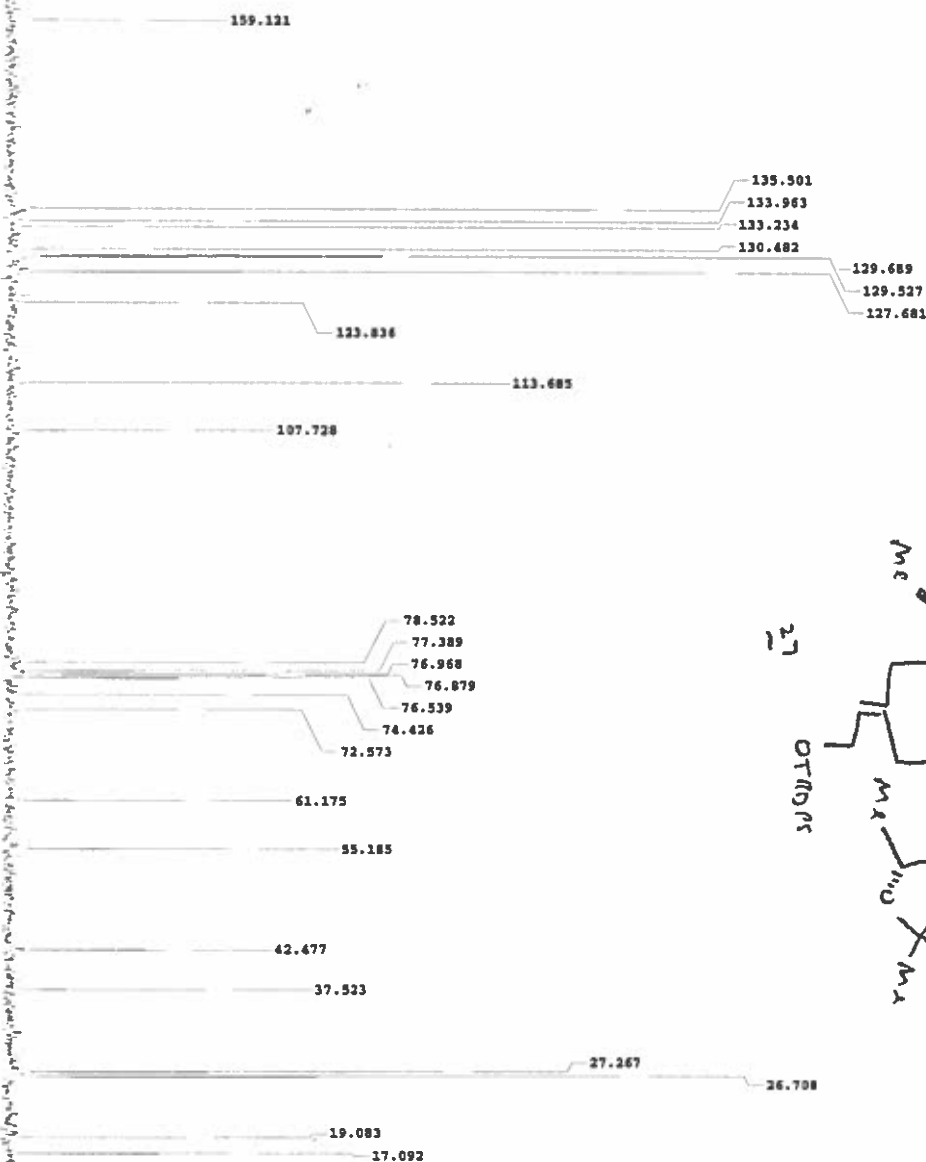




13C OBSTERVE

```
Solvent: CDCl3
Ambient temperature
Sample #7
Pile: 701
Queue name Feb25a
DATE Feb 25 01
INVOYA-300 "athos"
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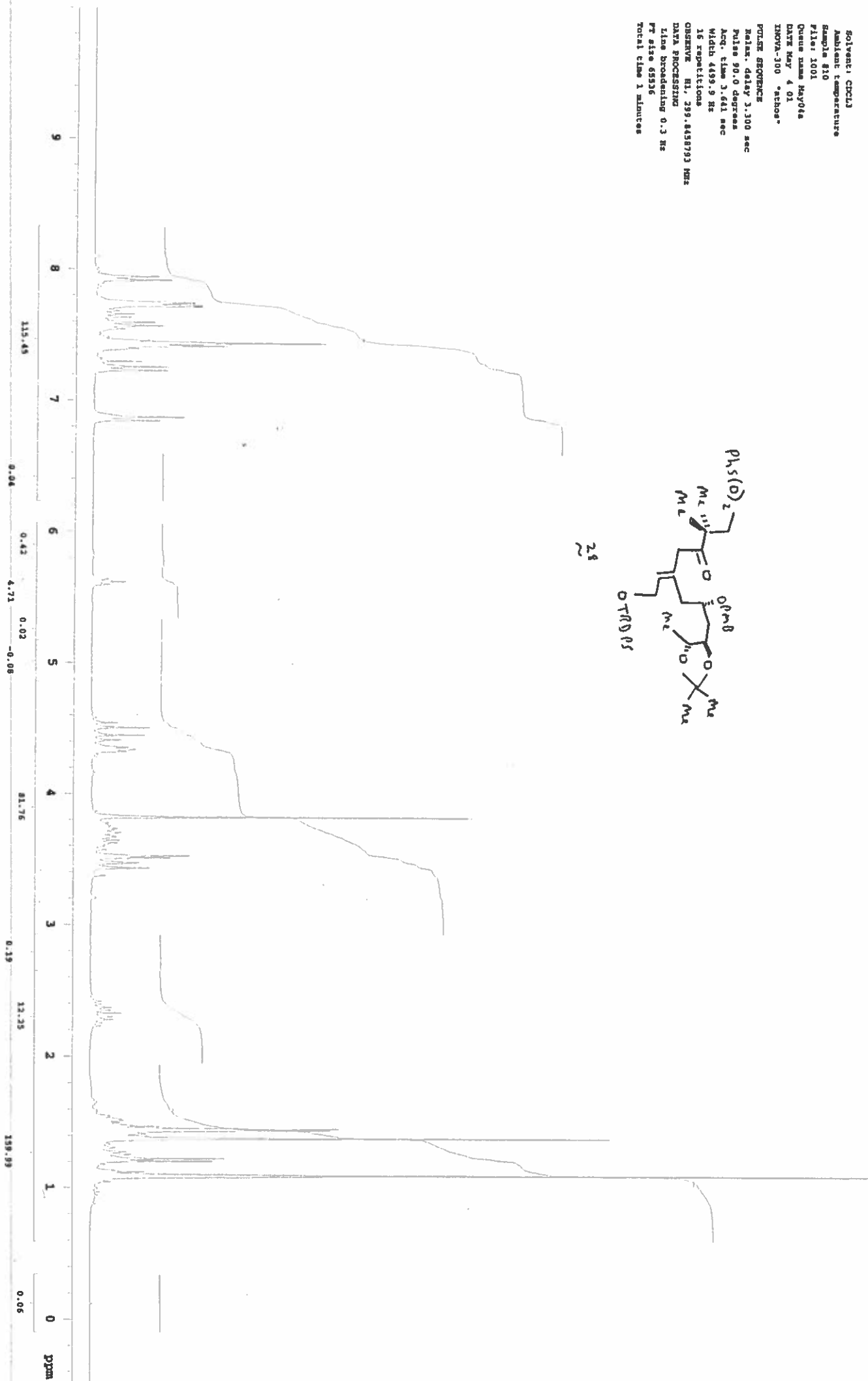
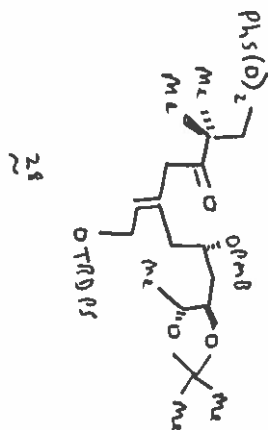
PULSE SEQUENCE  
 Pulse 45.0 degrees  
 Acq. time 1.68 sec  
 Width 30000.0 Hz  
 176 repetitions  
 DISCRIM. C13, 75.3963141 MHz  
 OBSERVE H1, 299.843785 MHz  
 Power 40 dB  
 continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 65536  
 spectral lines & mixtures



HEB031/1 (5-11)  
STANDARD IN OBSERVE

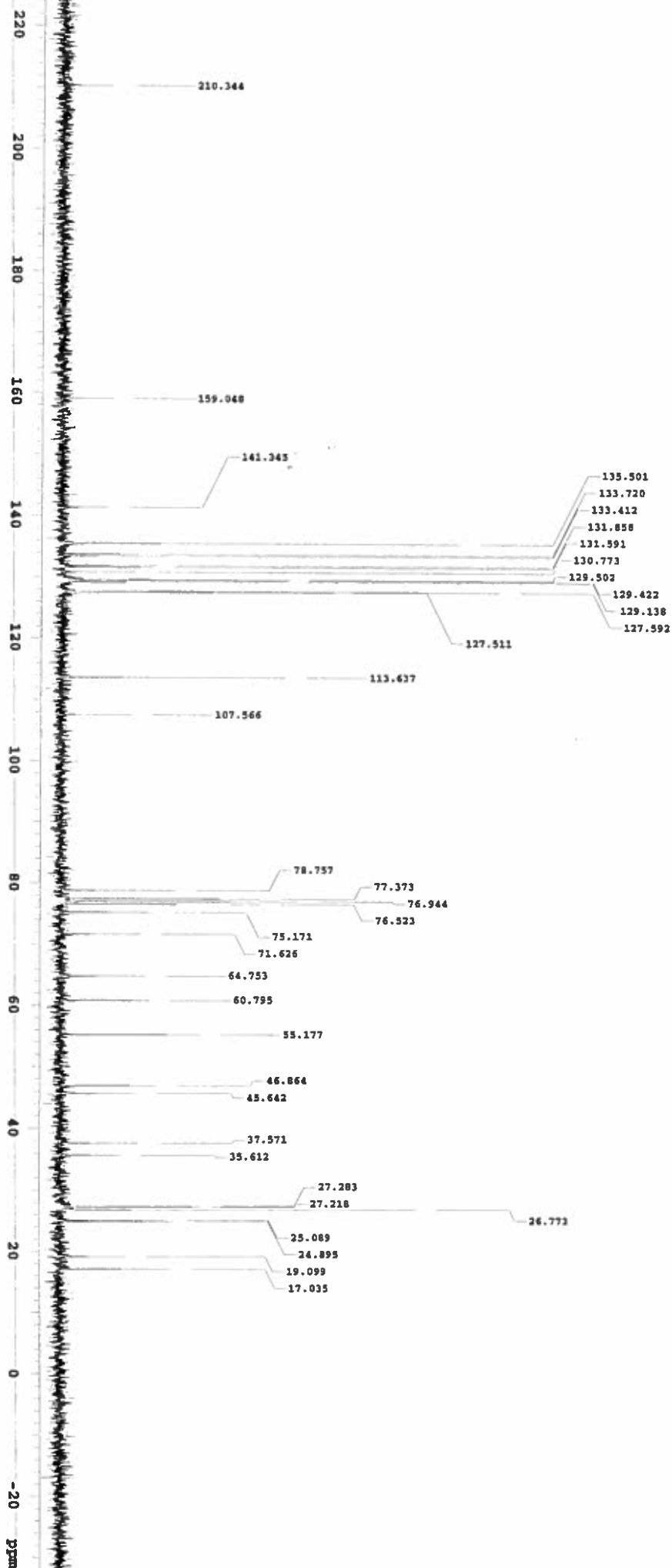
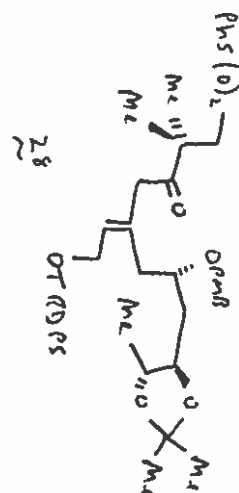
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #10  
File: 1001  
Queue name May06a  
Date May 4 01  
INOVA-300 "athos"

PTMR SEQUENCE  
Relax. delay 3.100 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions  
OBSERVE H1, 299.8458793 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
PT size 65536  
Total time 1 minutes



MR1011/L (5-11)  
13C OBSERVE

Solvent: CDCl3  
Ambient temperature  
Sample #32  
File: 1101  
Queue name May04a  
Date May 4 01  
INSTR-100 "achos"  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.618 sec  
Width 20000.0 Hz  
256 repetitions  
OBSERVE CH, 75.396141 MHz  
DECOUPLE H1, 299.847785 MHz  
Power 40 dB  
continuously on  
MALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
ST size 65536  
Total time 7 minutes

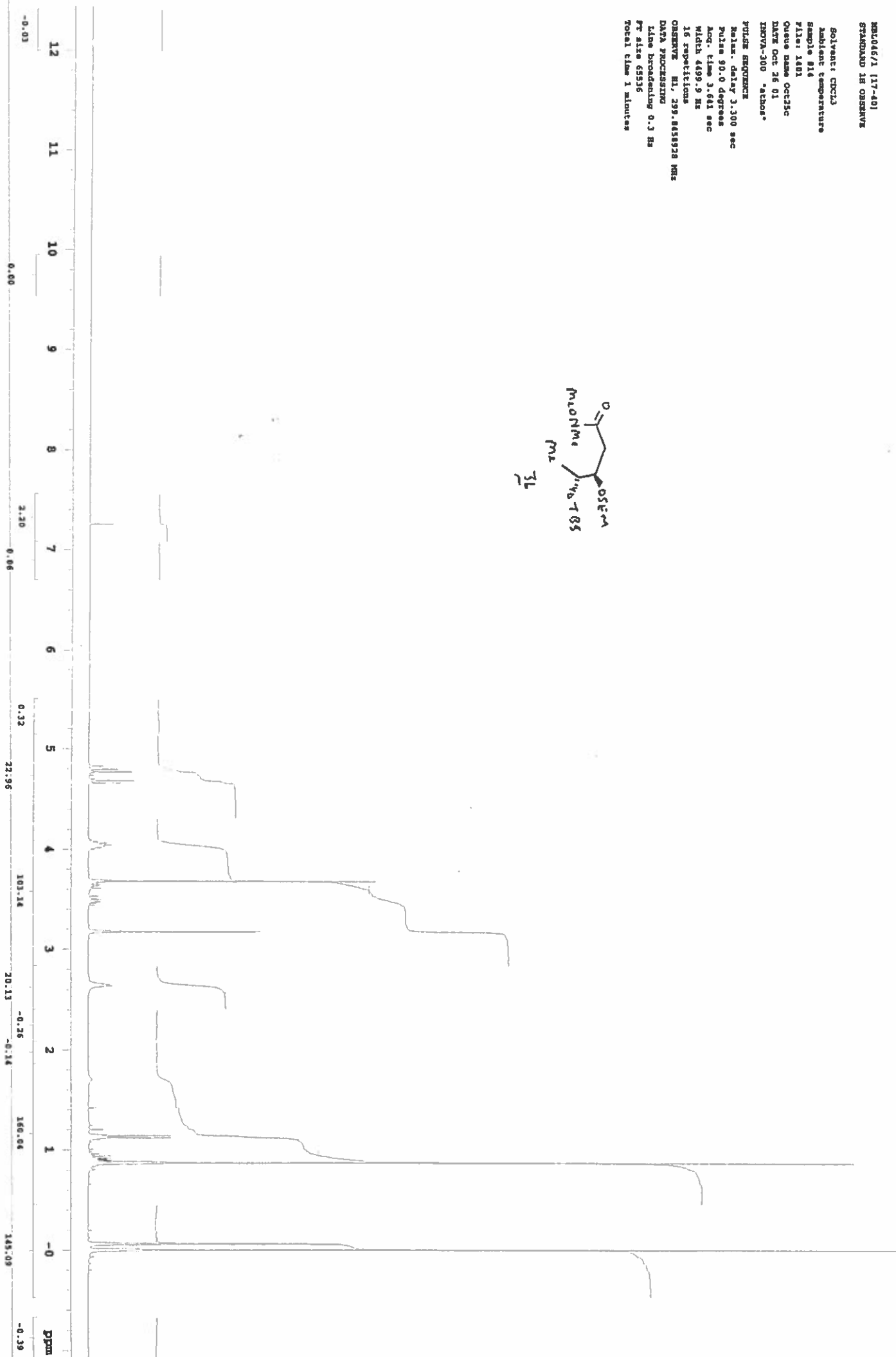
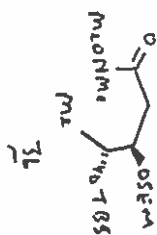


MR046/1 (17-40)  
STANDARD 1H OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #14  
File: 1401  
Queue name Oct15c  
Date Oct 26 01  
INSTR-300 "atom"

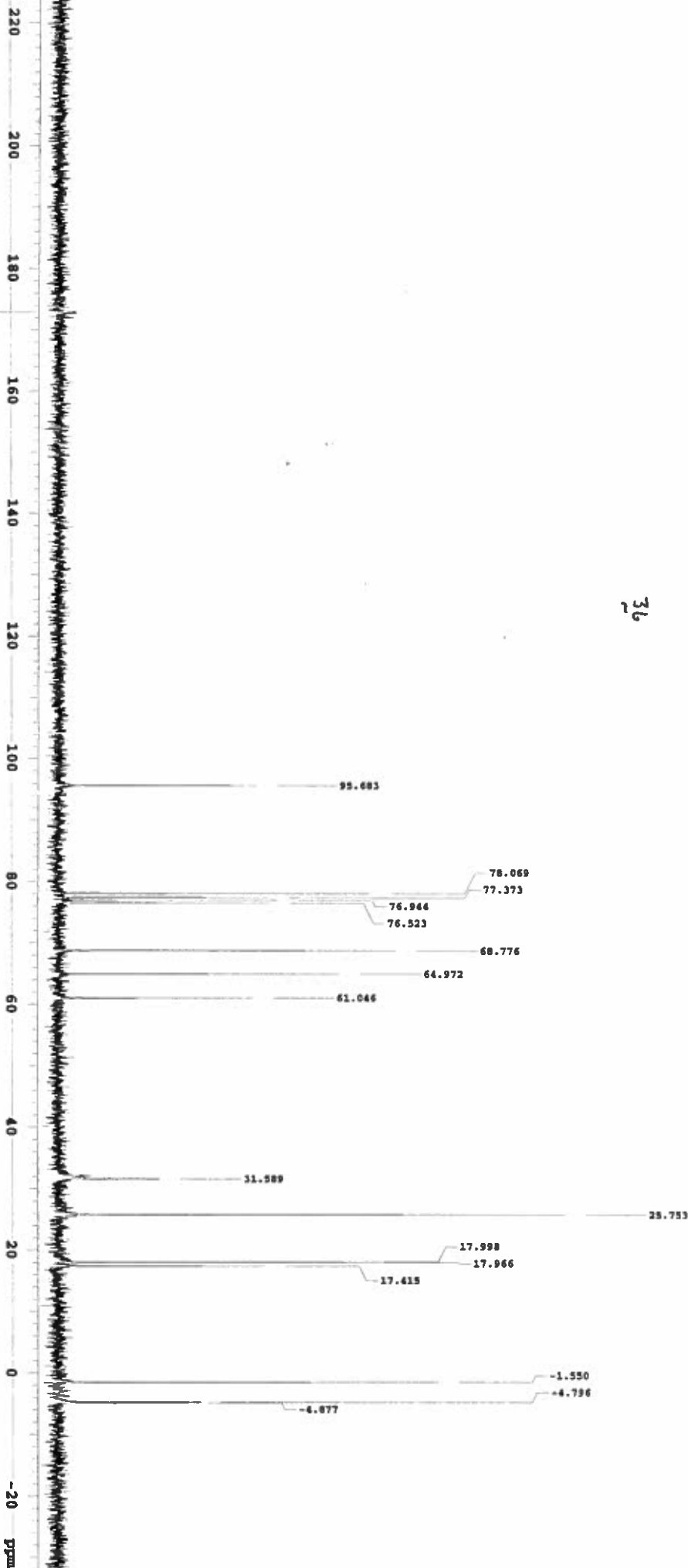
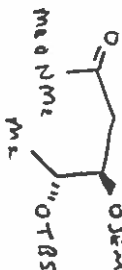
PULSE PROGRAM  
Pulse delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions

OBSERVE H1, 299.845828 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
PT size 65536  
Total time 1 minutes



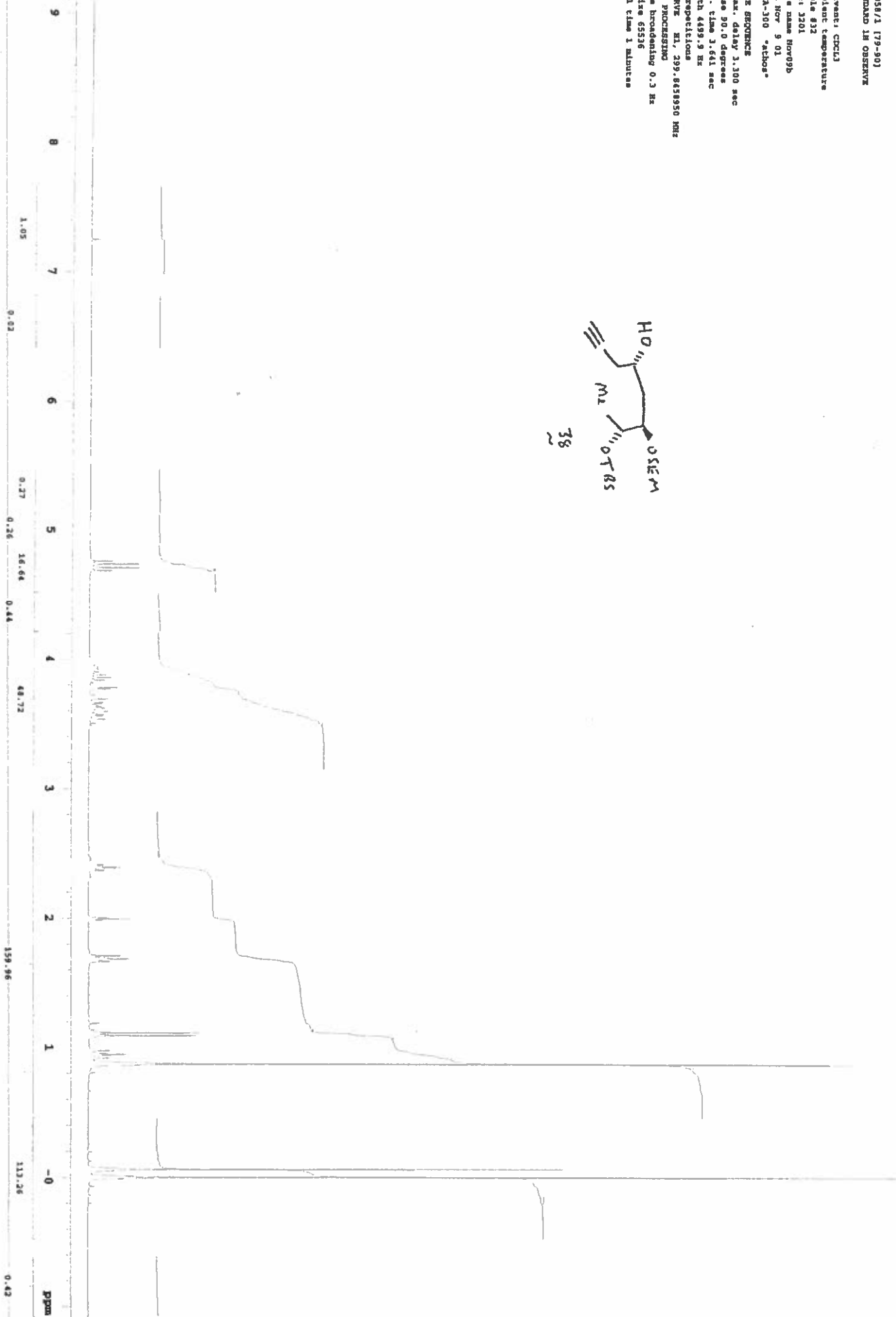
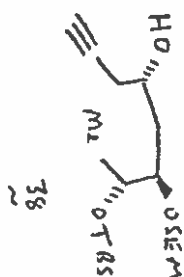
MBL056/3  
1JC OBSERVE

Solvent: CDCl3  
Ambient temperature  
Sample #11  
File: 1101  
Queue name Feb06a  
DATE Feb 6 02  
INSTR-300 "athos"  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.518 sec  
Width 20000.0 Hz  
256 repetitions  
OBSERVE C13, 75.393141 MHz  
DECOUPLE H1, 299.8473785 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 7 minutes



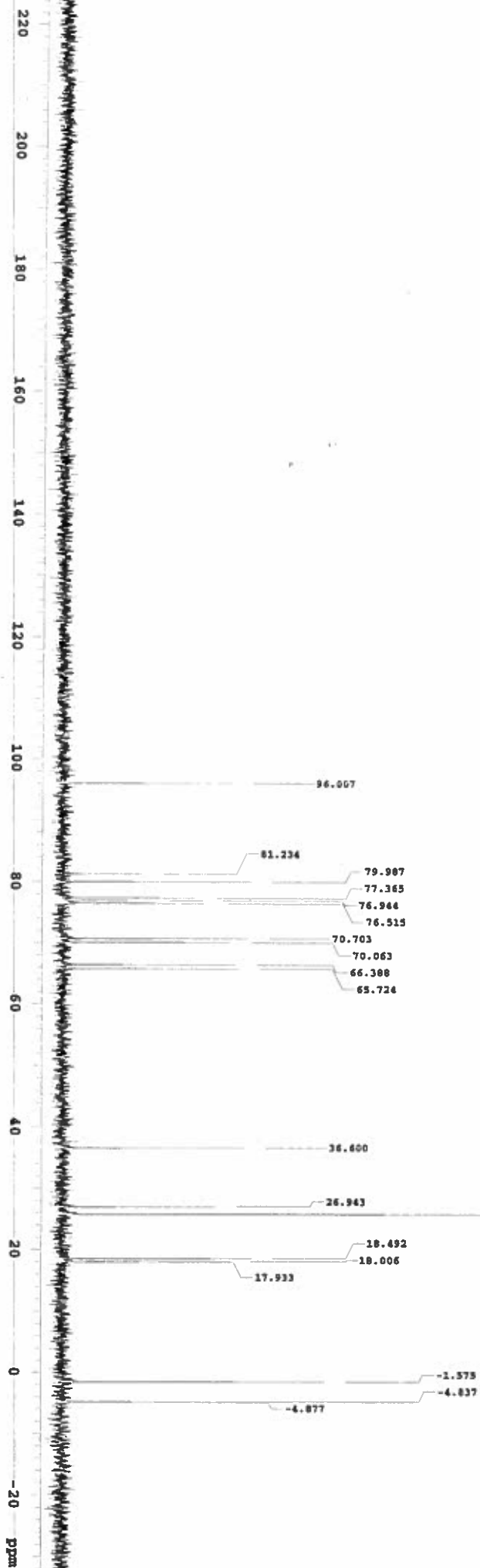
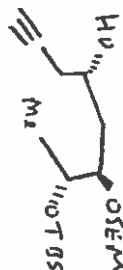
NM1058/1 (179-90)  
STANDARD 1H OBSERVE

Solvent: CDCl3  
Ambient temperature  
Sample #32  
File: 3201  
Queue name Nov09b  
Date Nov 9 01  
INOVA-100 "athos"  
PULSE SEQUENCE  
Relax. delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.641 sec  
Width 4499.9 Hz  
16 repetitions  
OBSERVE E1, 299.845850 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 3 minutes



HM058/1 (79-90)  
13C OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Sample #6  
File: 601  
Queue Name: 6011a  
Date Nov 11 01  
INOVA-100 "atbos"  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.638 sec  
Width 20000.0 Hz  
224 repetitions  
OBSERVE CH1, 75.3963141 MHz  
DECOUPLE H1, 299.8473785 MHz  
Power 40 dB  
continuously on  
MAGPIE-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 6 minutes



MBL075/1 (13-28)  
STANDARD IN OBSERVE

```

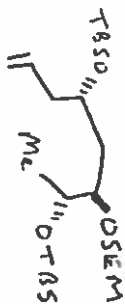
Solvent: CDCl3
Ambient temperature
Sample #22
File: 2201
Queue name Jan09b
Date Jan 9 02
INOVA-100 "athos"

```

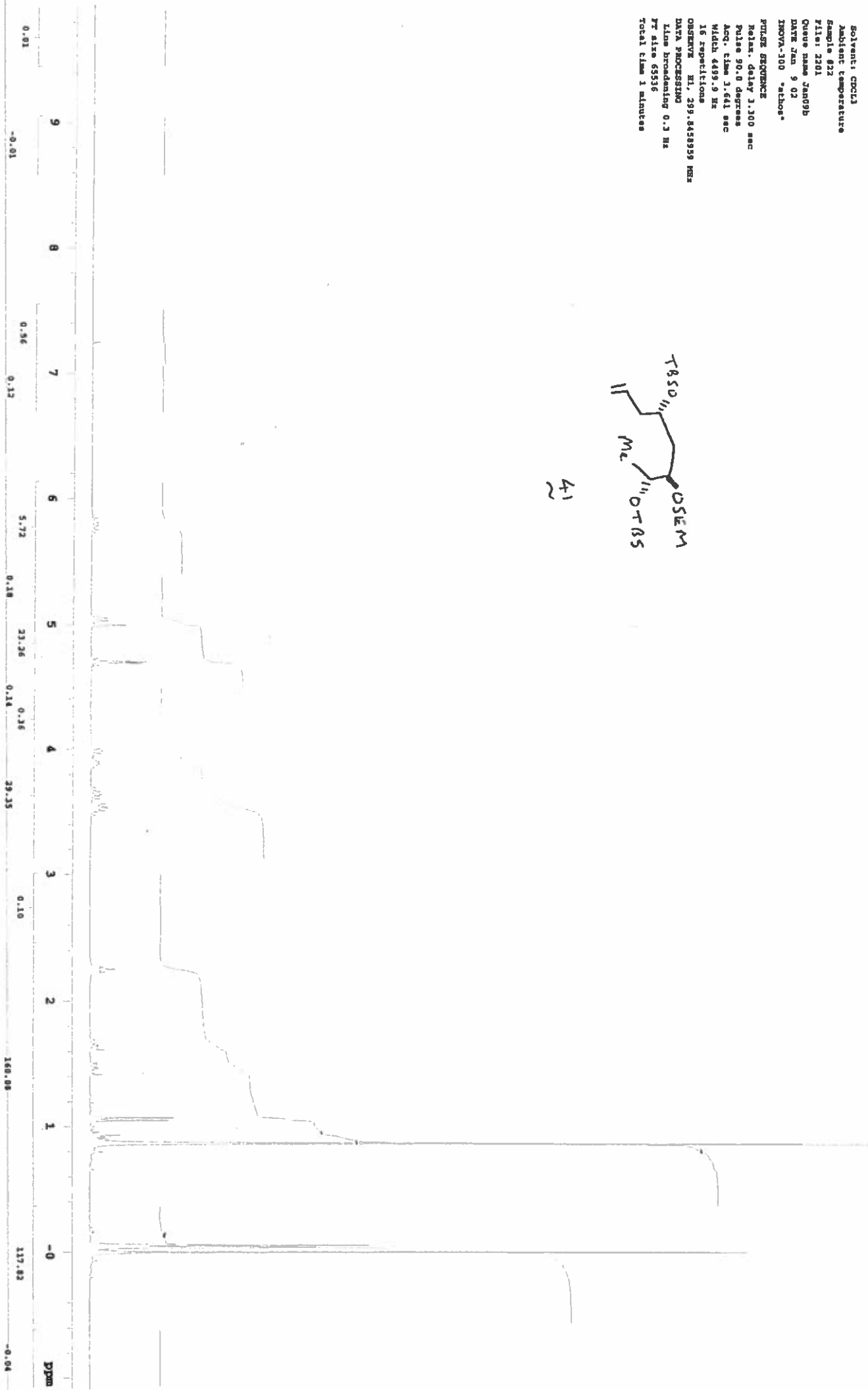
```

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4459.9 Hz
16 repetitions
OBSERVE H1, 209.8459359 MHz
DATA PROCESSING
Line broadening 0.3 Hz
Ft size 65536
Total time 1 minutes

```

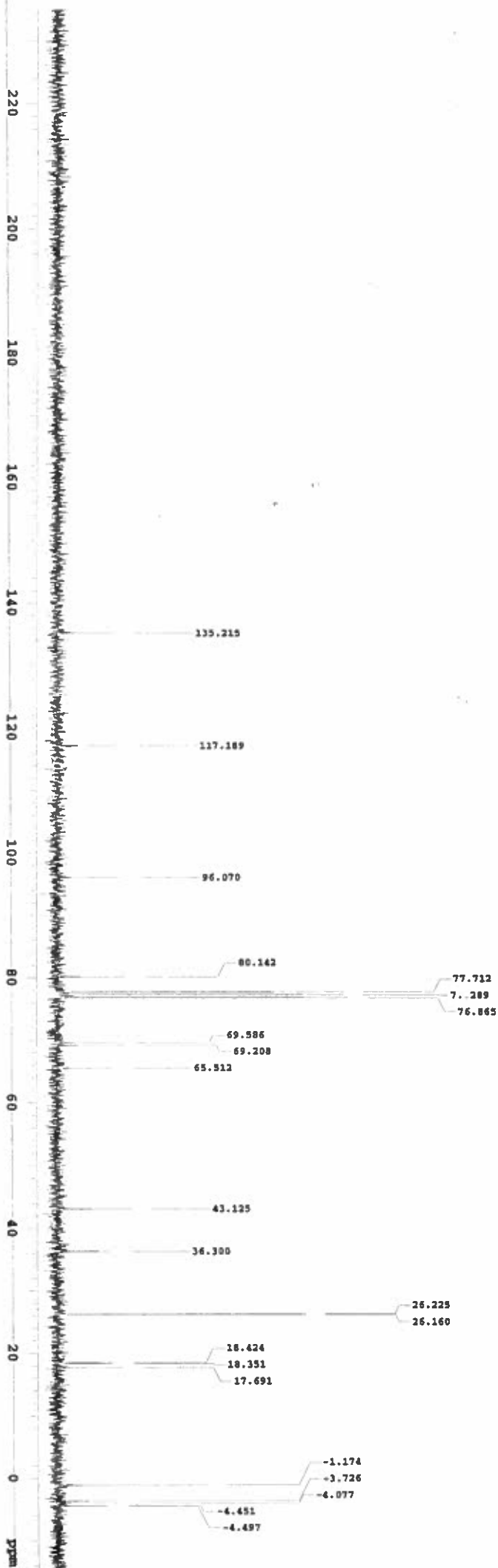
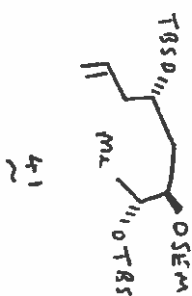


24





1

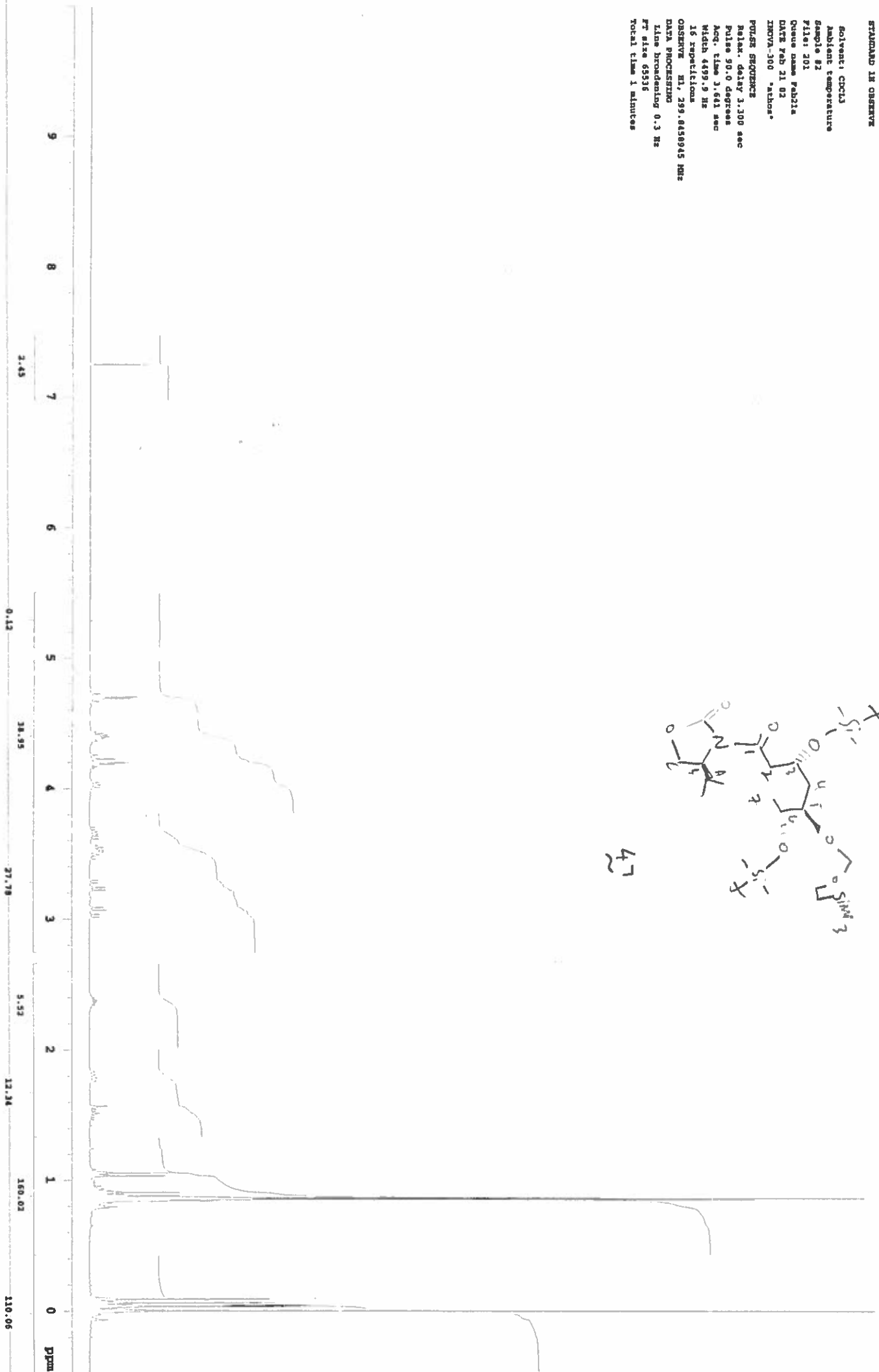
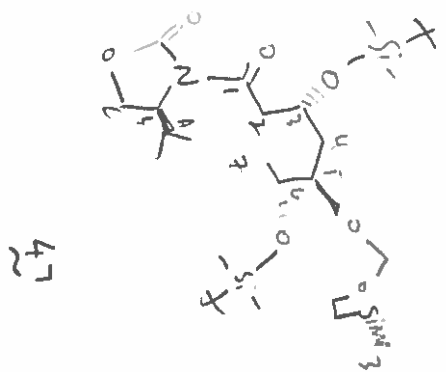


BD104/1 (3-7)  
STANDARD IN OBSERVE

Solvent: CCl<sub>4</sub>  
Ambient temperature  
Sample 82  
File: 201  
Queue name Feb21a  
DATE Feb 21 02  
INSTR 300 MHz

PULSE SEQUENCE  
Pulpr. delay 3.300 sec  
Pulse 90.0 degrees  
Acq. time 3.661 sec  
Width 4499.9 Hz  
16 repetitions

OBSERVE H1, 299.8458945 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 minutes



MBL084/1 (3-7)  
13C OBSERVE

Solvent: CDCl<sub>3</sub>  
Ambient temperature

Sample #3

Queue name Mar06b

IMPROVA-300 Authors

Pulse 45.0 d

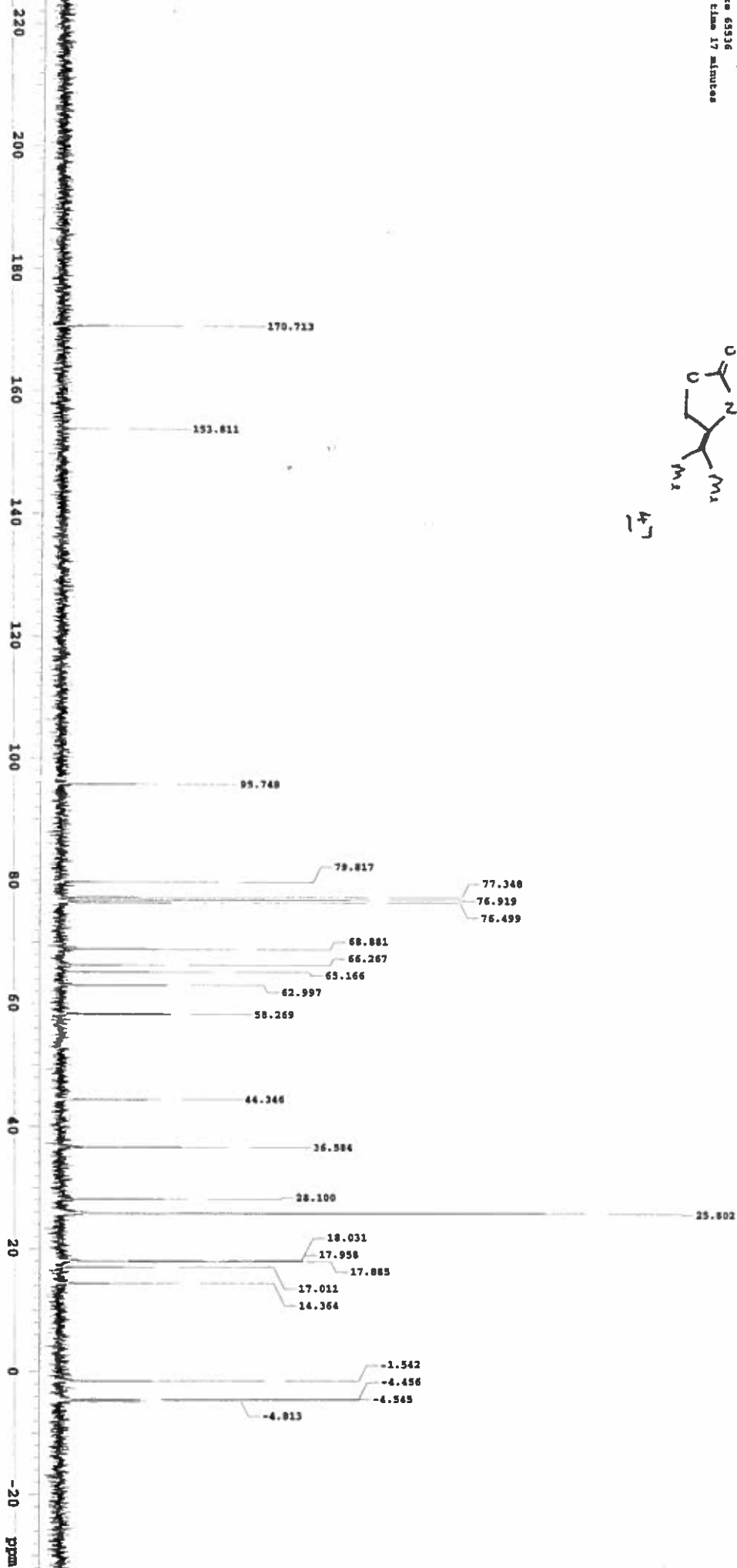
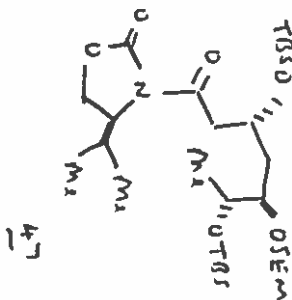
Pulse 45.0 degrees  
Acq. time 1.630 secWidth 20000.0 Hz  
656 repetitions

OBSERVE C13, 75.3963141 MRS  
DECORPUS H1, 399.0473705 MRS

Power 40 dB  
Continuously onWALTZ-16 modulated  
DATA PROCESSING

Line broadening 1.0 Hz

**Total time 17 minutes**



mb1 104/1 (49-59)  
STANDARD IN OBSERV

Solvent:  $\text{CDCl}_3$   
Ambient temperature

Sample #35  
File: 3501

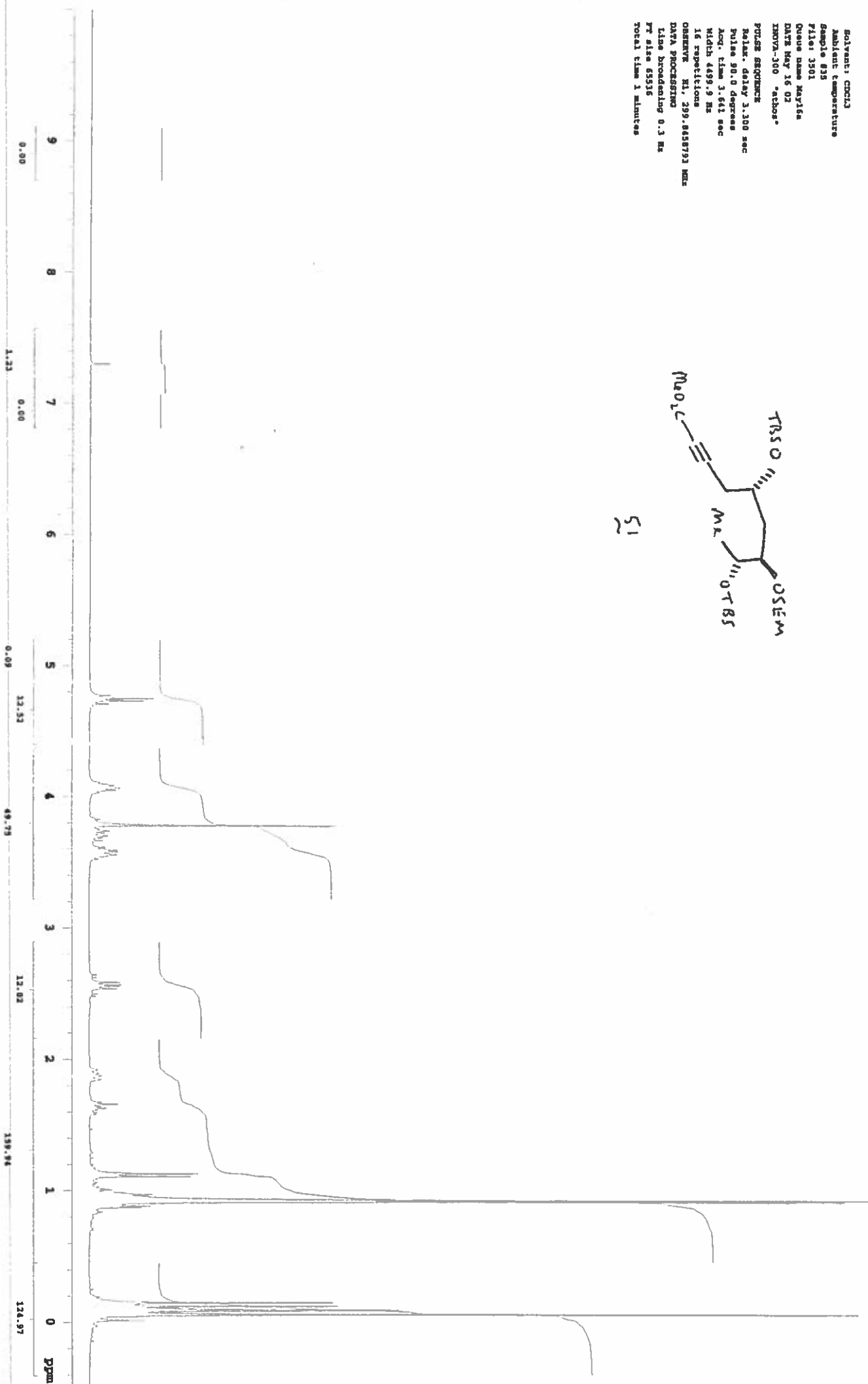
DATE MAY 16 02  
INVOY-300 "ACHOS"

**PULSE REQUEST**  
Ref: 4314 3 100 201

Pulse 90.0 degrees  
Acq. time 3.641 secWidth 4699.9 m  
16 repetitions

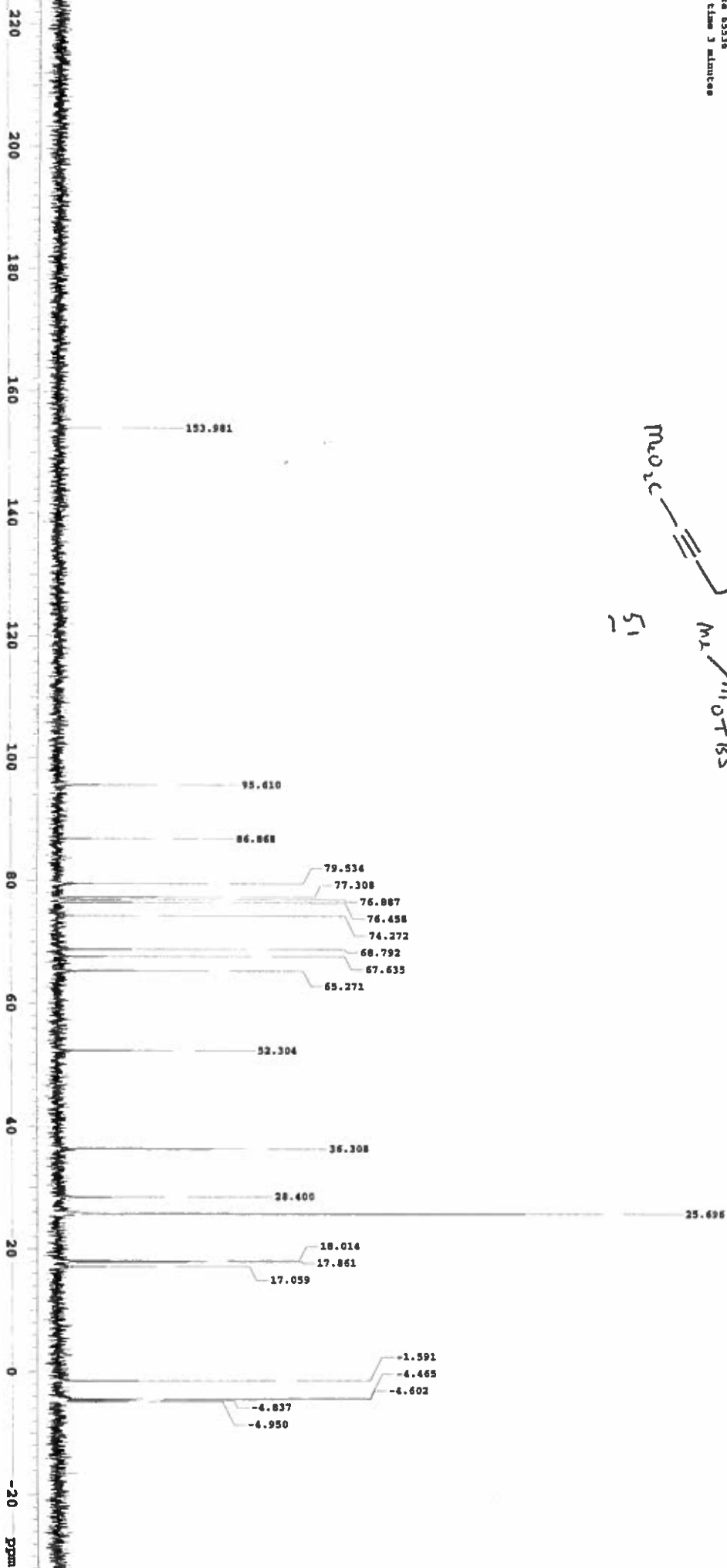
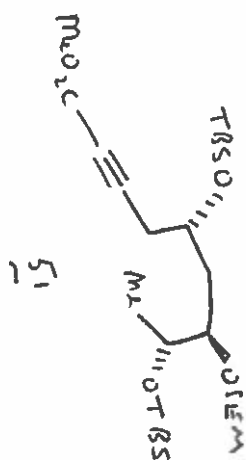
OBSERVE HL, 299.8658793 MHz  
DATA PROCESSING

PT dia 65536  
Total time 1 minutes



mbi 104/1 (49-59)  
1JC OBSERVE

Solvent: CDCl3  
Ambient temperature  
Sample 827  
File: 2701  
Queue name May27b  
DATE May 28 02  
INSTR-300 "athos"  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 1.618 sec  
Width 20000.0 Hz  
144 repetitions  
OBSERVE C13, 75.396141 MHz  
DECOUPLE H1, 299.847185 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
PT size 65536  
Total time 3 minutes

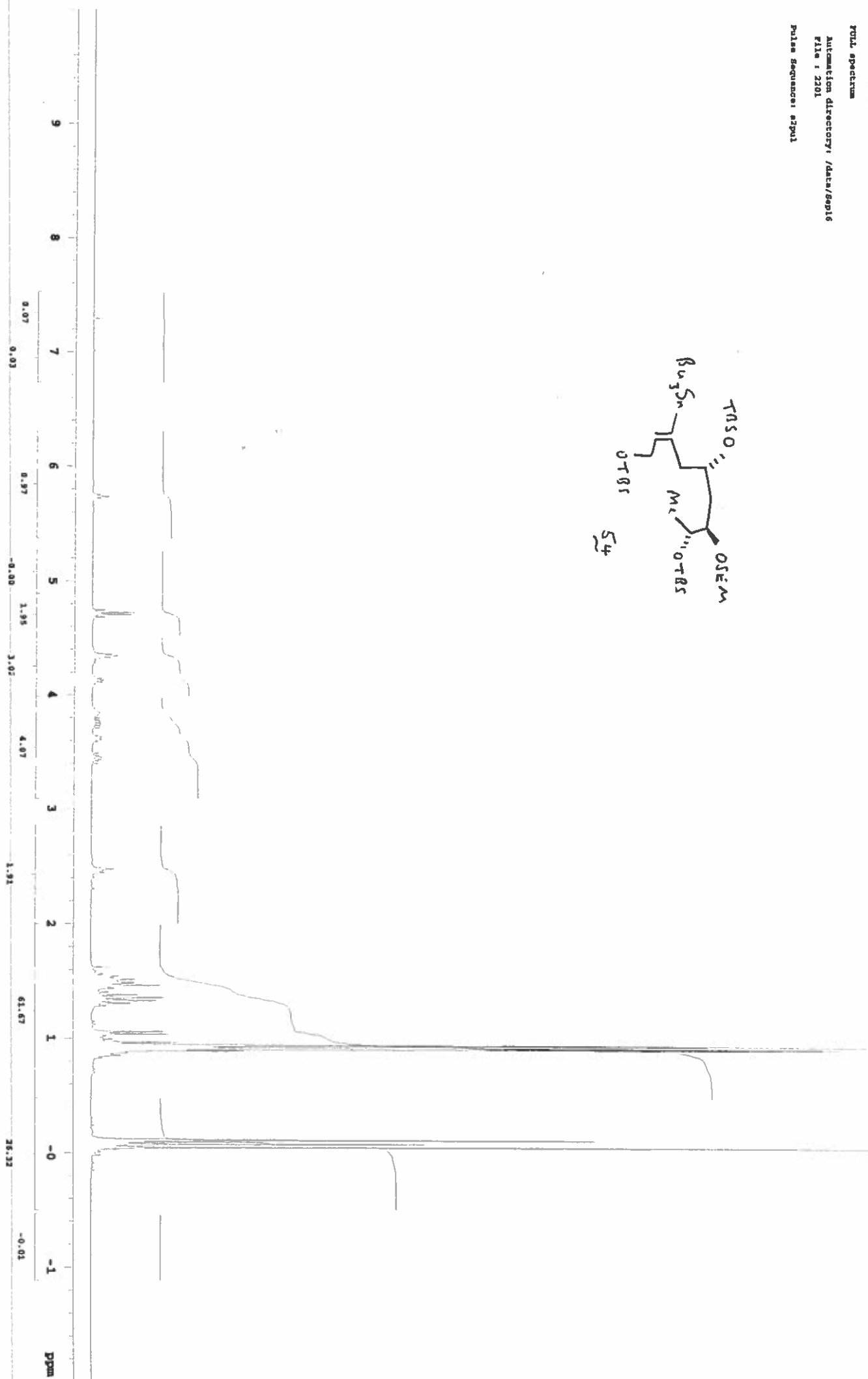
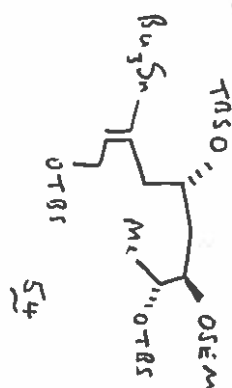


mbi 114/1 pure  
STANDARD IR OBSERVE

FTIR spectrum

Automation directory: /data/sep16  
File : 2201

Pulse Sequence: aqpu1



mb1 114/1 pure  
STANDARD 1R OBSERVE

Automation directory: /data/sep16  
File: 2203

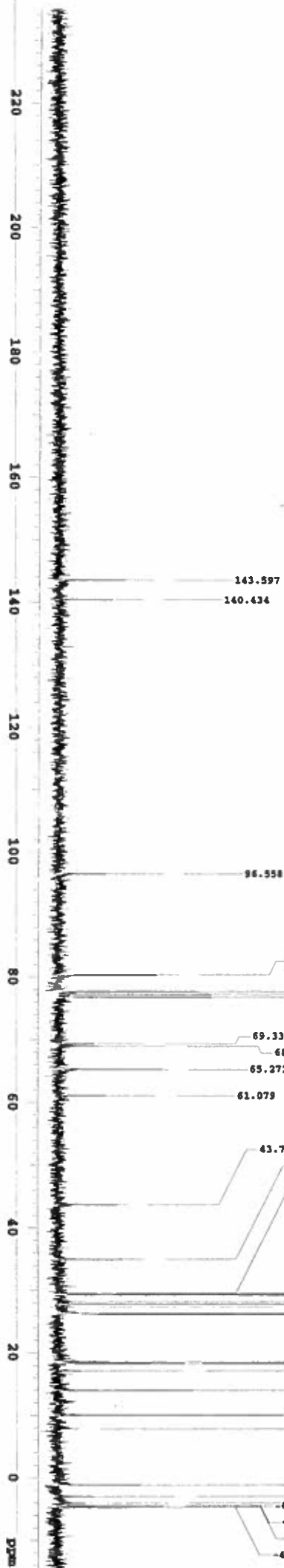
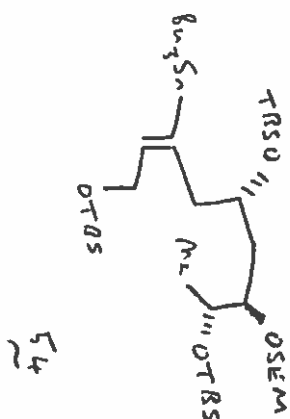
Pulse Sequence: zgpg1

Solvent: cdcl3  
Ambient temperature

Sample 822

File: 2203  
INVTX-300 "entry"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18050.1 Hz  
256 repetitions  
OBSERVE C13, 75.392883 MHz  
DECOUPLE M1, 299.847786 MHz  
Power 17 dB  
continually on  
NAUT-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 12 min, 2 sec



143.597  
140.434

96.558

80.321

77.681  
77.258  
76.835

69.334  
68.964  
65.271

61.079

43.713  
34.939  
29.502

29.373  
29.247  
28.175  
27.782  
27.389

26.263  
26.244  
26.126  
18.618  
18.397  
18.313

18.286  
17.100  
13.971  
9.981  
7.799

-1.204  
-3.032  
-4.127

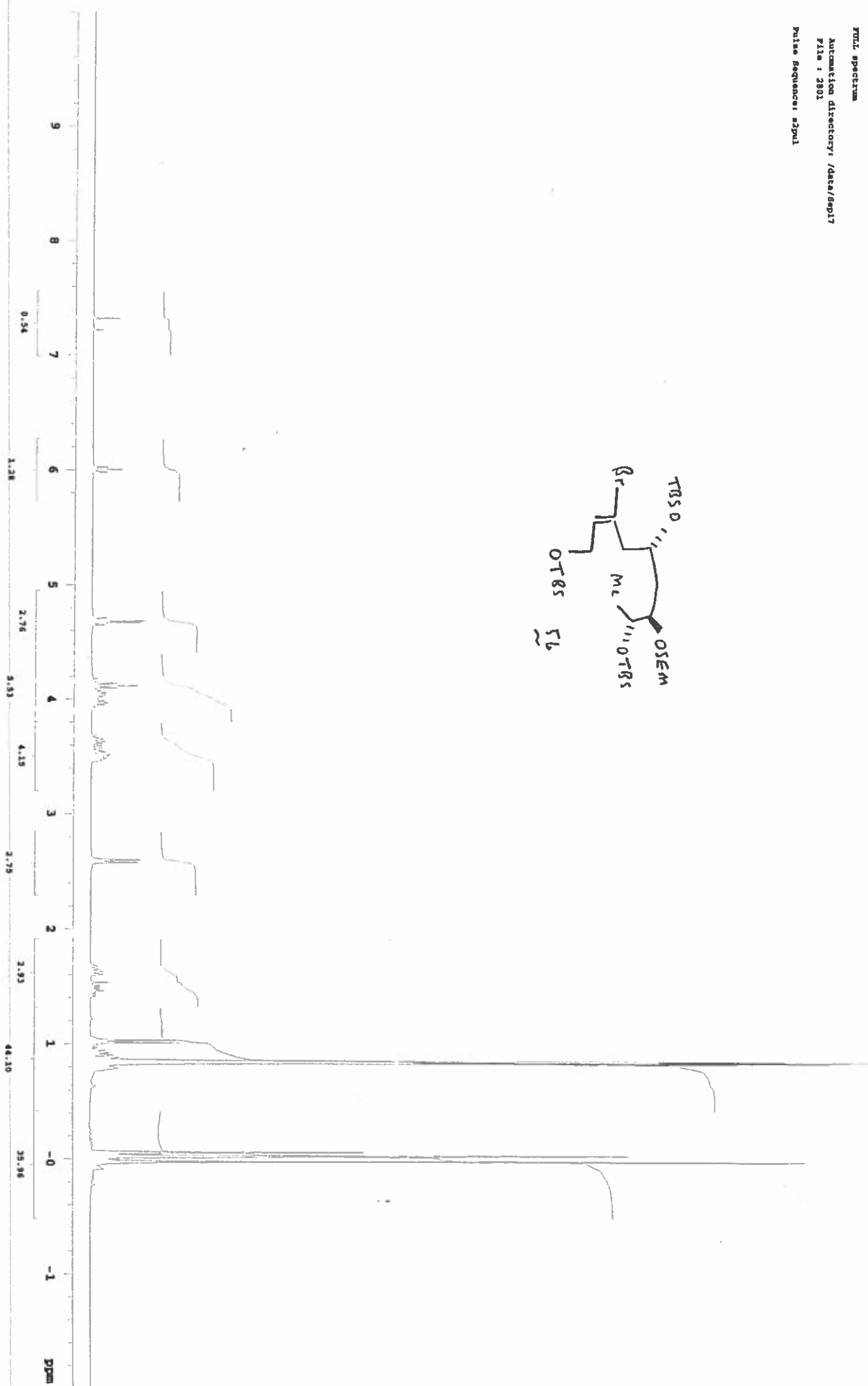
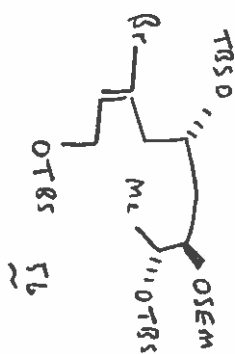
-4.497  
-4.569  
-4.741  
-4.768

ab1 115/1 pure  
STANDARD 1F OBSERVE

FULL SPECTRUM

Automation directory: /data/sep17  
File : 2801

Pulse sequence: zgpg1



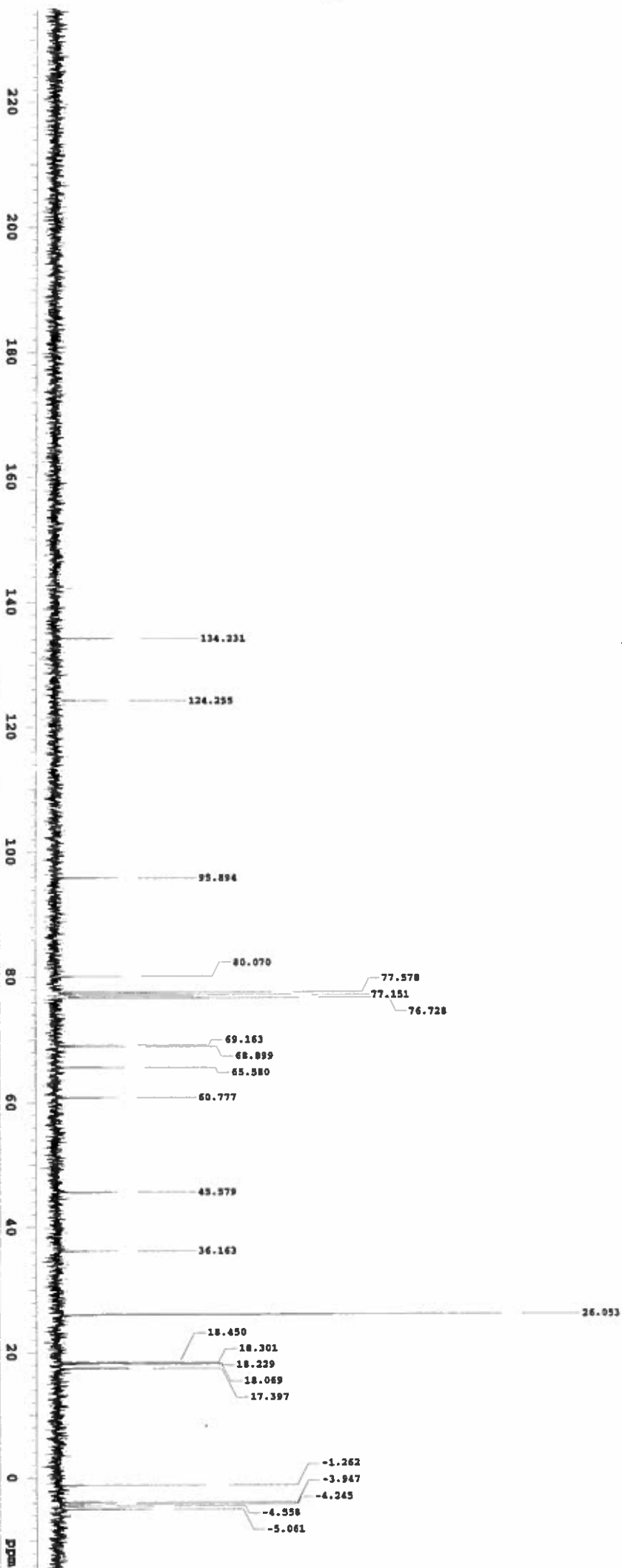
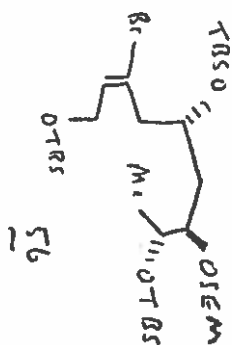


mbj 115/1 pure  
STANDARD IR OBSERVE

Automation directory: /data/seq17  
File : 2803

Pulse Sequence: szpul  
Solvent: cdcl3  
Ambient temperature  
Sample #28  
File: 2803  
INOVA-300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18850.1 Hz  
256 repetitions  
OBSERVE C13, 75.3962883 MHz  
DECOUPLE H1, 299.8471786 MHz  
Power 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 12 min, 2 sec



mb1 120/5  
STANDARD IN OBSERVE

Automation directory: /data/rundata/Feb26  
File : 2004

Pulse Sequence: #2pul

Solvent: cdcl<sub>3</sub>

Ambient temperature

FILE: 2004

**INNOVA-300** **•** **TRACER**

Relax, delay 1.000 sec

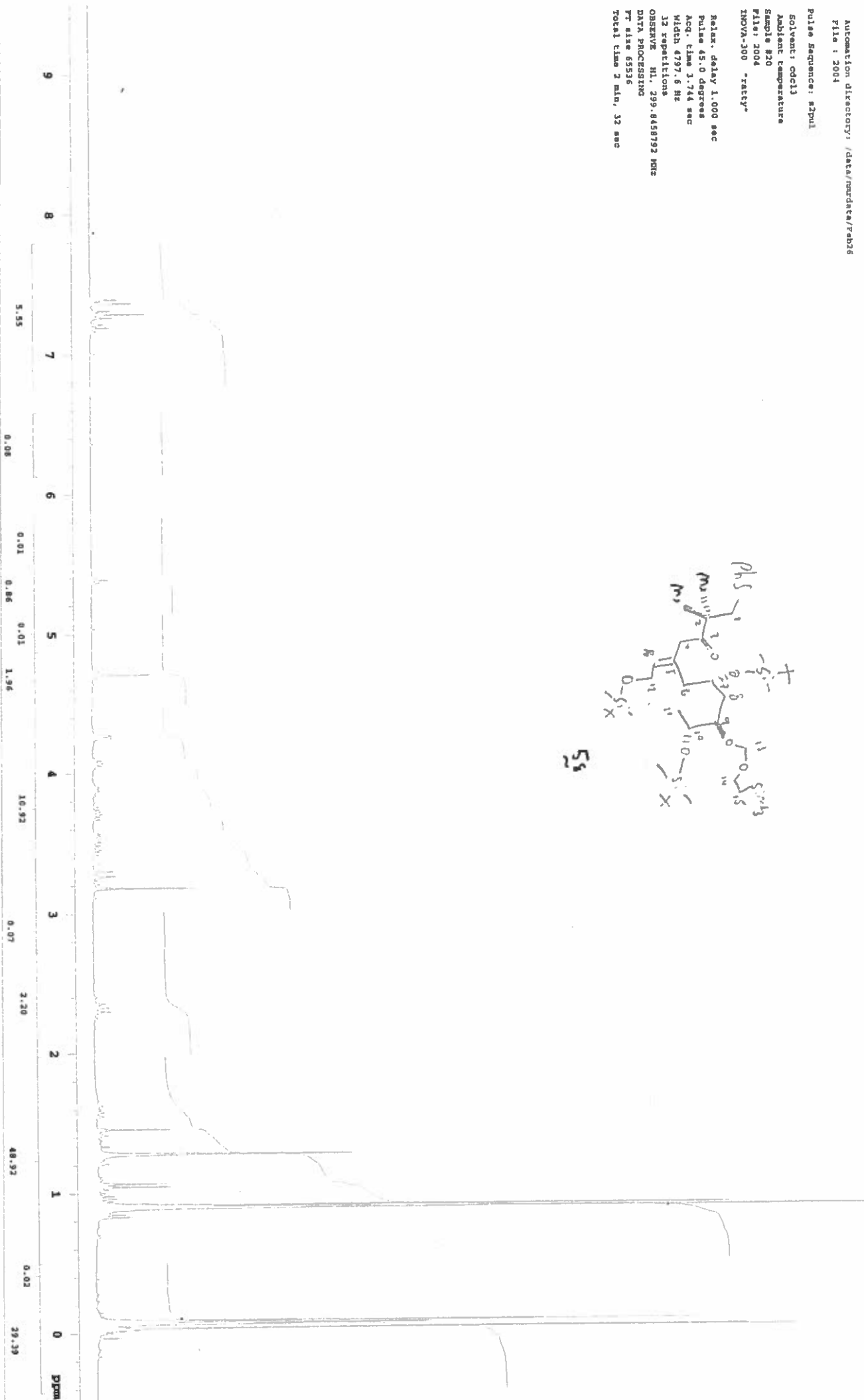
[illegible]

Width 4797.6 ft

25 APR 1968  
OBSERVE HL, 299.0450792 MHz

## DATA PROCESSING

14 4138 03336

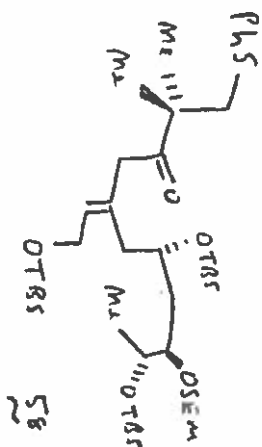


abl 120 pure  
STANDARD 1H OBSERVE

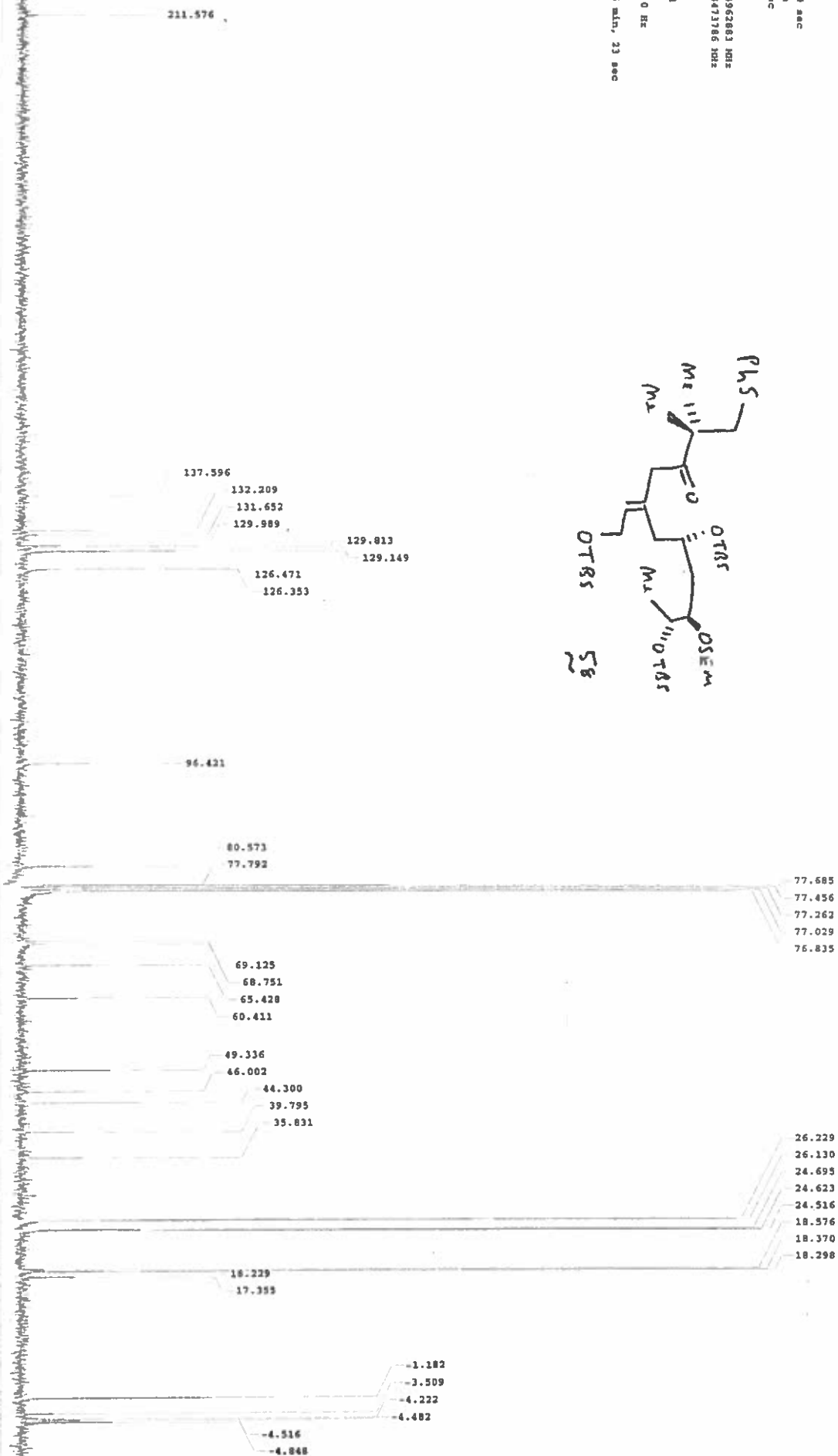
Automation directory: /data/mwdata/feb21  
File: 0905

Pulse Sequence: mZpul  
Solvent: cdcl3  
Ambient Temperature  
Sample 89  
File: 0905  
INOVA-300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18850.1 Hz  
2048 repetitions  
OBSERVE C13, 75.3962883 MHz  
DECOUPLE H1, 299.647186 MHz  
Power 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 1 hr, 36 min, 23 sec



220 200 180 160 140 120 100 80 60 40 20 0 ppm

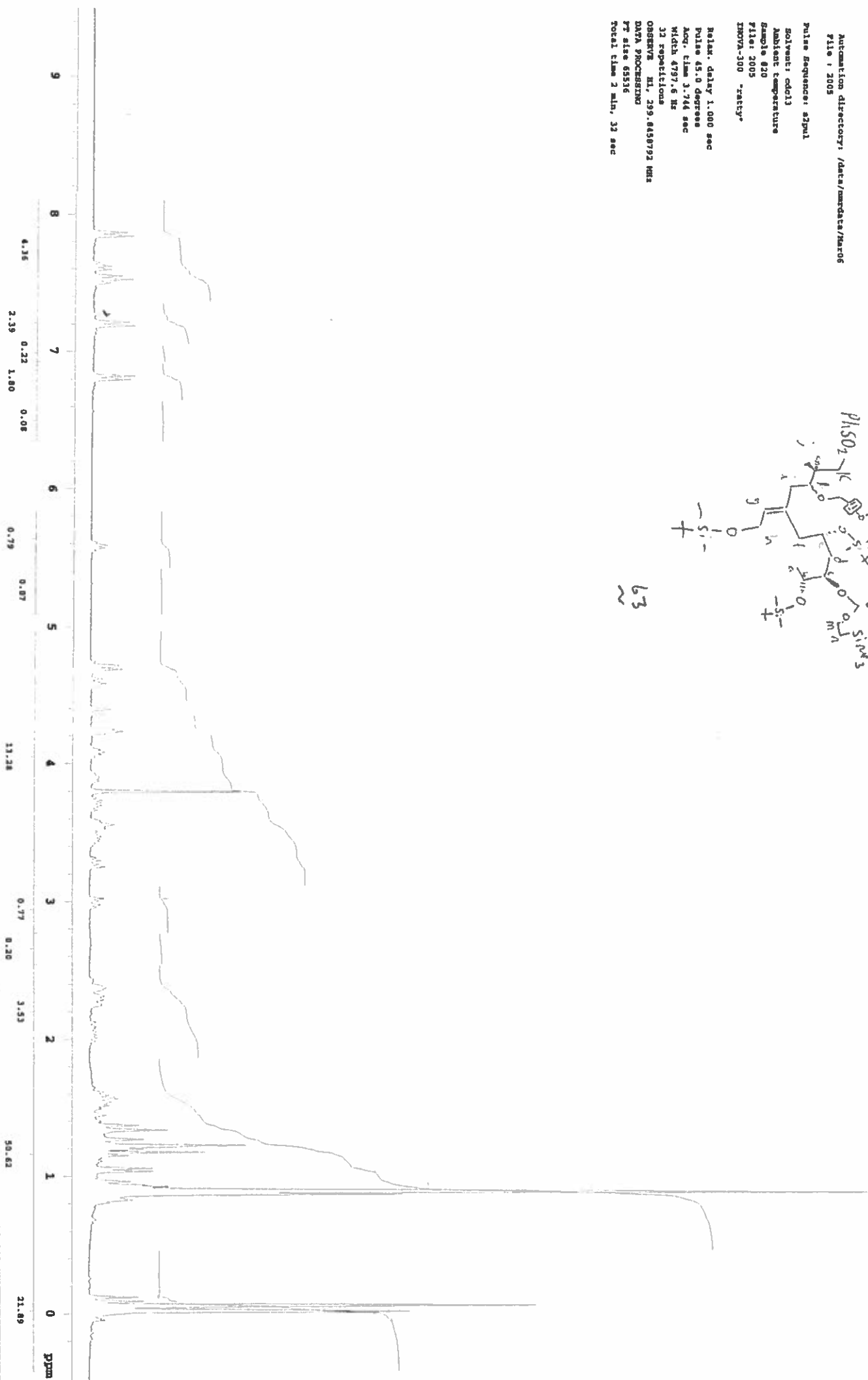
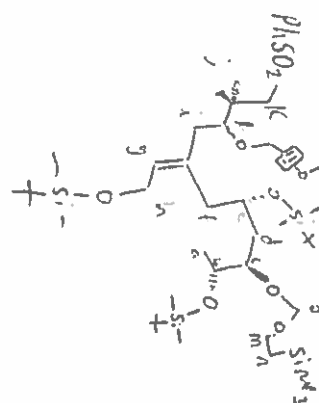


AD155/2  
STANDARD IN OBSERVE

Automation directory: /data/mwdata/Nov06  
File: 2005

Pulse sequence: zgpg30  
Solvent: cdcl3  
Ambient temperature  
Sample: 820  
File: 2005  
INVT-300 "raty"

Pulse delay: 1.000 sec  
Pulse: 45.0 degree  
Acq. time: 3.744 sec  
Width: 4797.6 Hz  
32 repetitions  
OBSERVE: H1, 299.8450792 MHz  
DATA PROCESSING  
FF else 65536  
Total time: 2 min, 32 sec



Matthew Ball

NBL 155

CDCl<sub>3</sub>

13C 75MHz

Inova 300

Portos(27)

10.12.03

1103024c

Pulse Sequence: zgpg30

Solvent: cdcl<sub>3</sub>

Ambient temperature

INOVA-300 "portos"

Pulse 109.1 degrees

Acq. time 1.250 sec

Width 20000.0 Hz

47701 repetitions

OBSERVE C13, 75.4610084 MHz

DECOUPLE H1, 300.1045968 MHz

Power 43 dB

continuously on

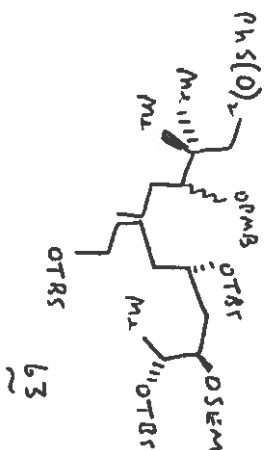
WALTZ-16 modulated

DATA PROCESSING

line broadening 1.0 Hz

FT size 65536

Total time 369 hr, 40 min, 32 sec



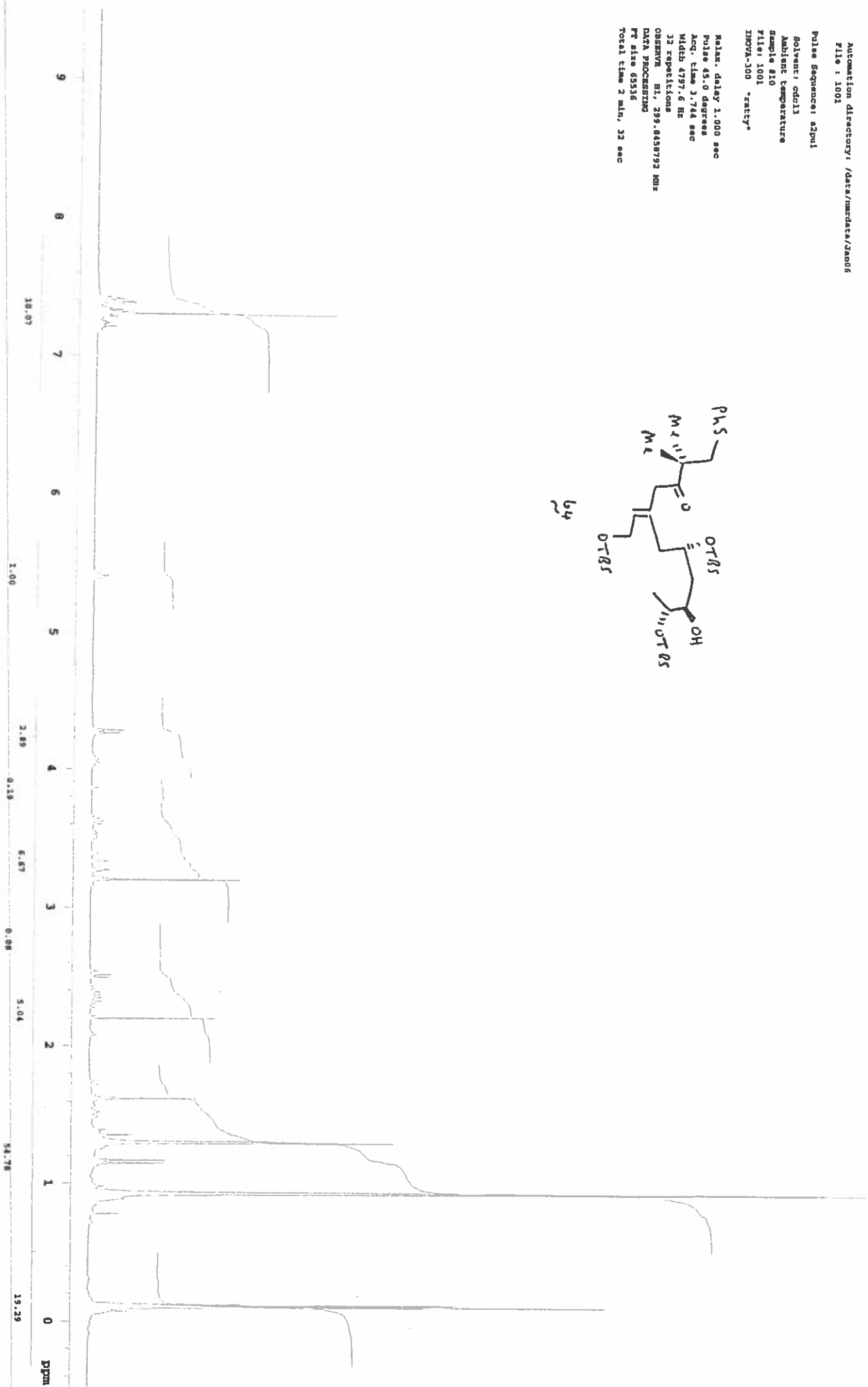
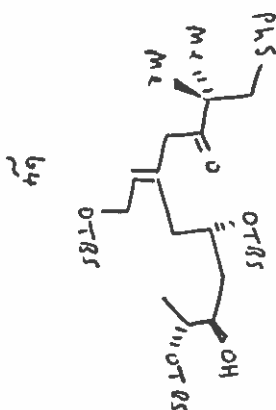
160 140 120 100 80 60 40 20 ppm

mb1 145  
STANDARD 1B OBSERVE

Automation directory: /data/nmrdata/jan06  
File: 1001

Pulse Sequence: zgpg30  
Solvent: cdcl3  
Ambient temperature  
Sample #10  
File: 1001  
INSTR: spect

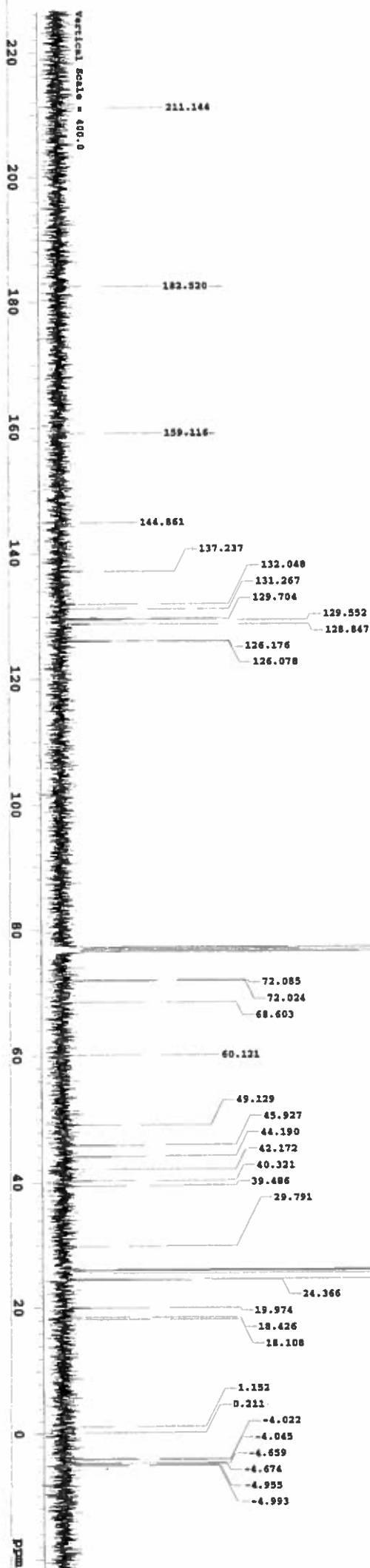
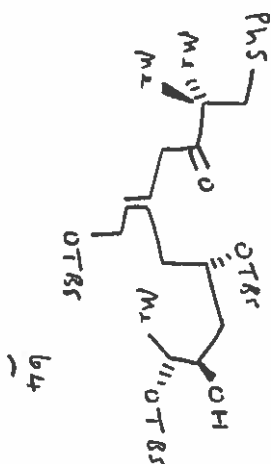
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 3.744 sec  
Width 4797.6 Hz  
32 repetitions  
OBSERVE H1: 299.8458792 MHz  
DATA PROCESSING  
F2 size 65536  
Total time 2 min, 32 sec



Mact Ball  
 KML 145  
 cdc13  
 Inova 400(GS)  
 Aromals  
 13C 100MHz  
 07.01.04  
 0104007C

Pulse Sequence: zgpg30  
 Solvent: cdcl3  
 Ambient temperature  
 INOVA-400 "aromals"

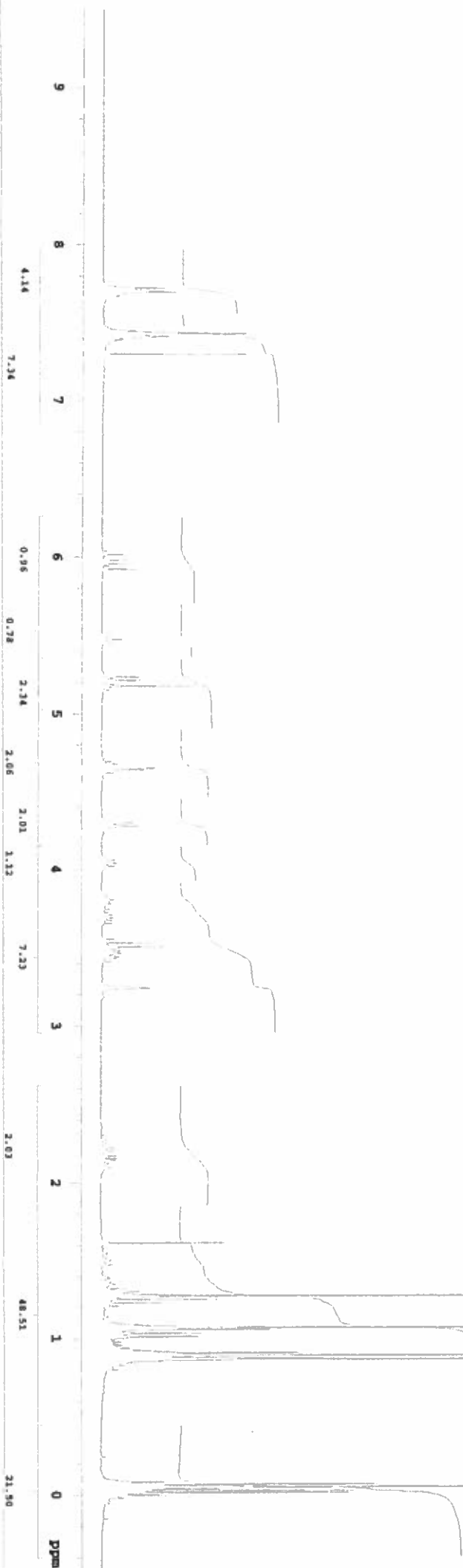
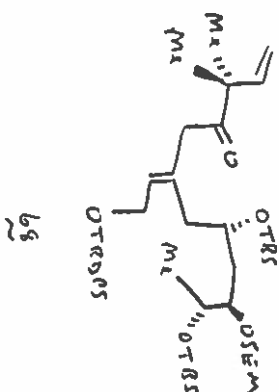
Pulse 35.0 degrees  
 Acq. time 1.31 sec  
 Width 25000.0 Hz  
 14468 repetitions  
 OBSERVE C13, 100.565308 MHz  
 DECOUPLE H1, 399.959752 MHz  
 Power 39 dB  
 continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 65536  
 Total time 366 hr, 32 min, 34 sec



Automation directory: /data/nmrdata/8621  
File: 2401

Pulse Sequence: zgpg30  
Solvent: cdcl3  
Ambient temperature  
Sample: 824  
File: 2401  
INOVA-300 "raccy"

Relax. delay: 1.000 sec  
Pulse: 45.0 degrees  
Acq. time: 3.744 sec  
Width: 4797.6 Hz  
16 repetitions  
OBSERVE: H1, 299.8458792 MHz  
DATA PROCESSING  
FT size: 65536  
Total time: 1 min, 16 sec





HS2  
13C NMR

pad-2 run with fluid0 before acquisition  
Automation directory: /data/mwdata/sep29  
File: 4001

Pulse sequence: zgpg

Solvent: cdcl3

Date Sep 29 2004

Sample 860

File: 4001

INSTR: 400 "rtecy"

Relax. delay 1.000 sec  
Pulse 45.0 degrees

Acq. time 1.015 sec

Width 18850.1 Hz

256 repetitions

OBSERVE CL3, 75.2962883 MHz

DECOUPLE H1, 299.8473786 MHz

Power 17 dB

continuously on

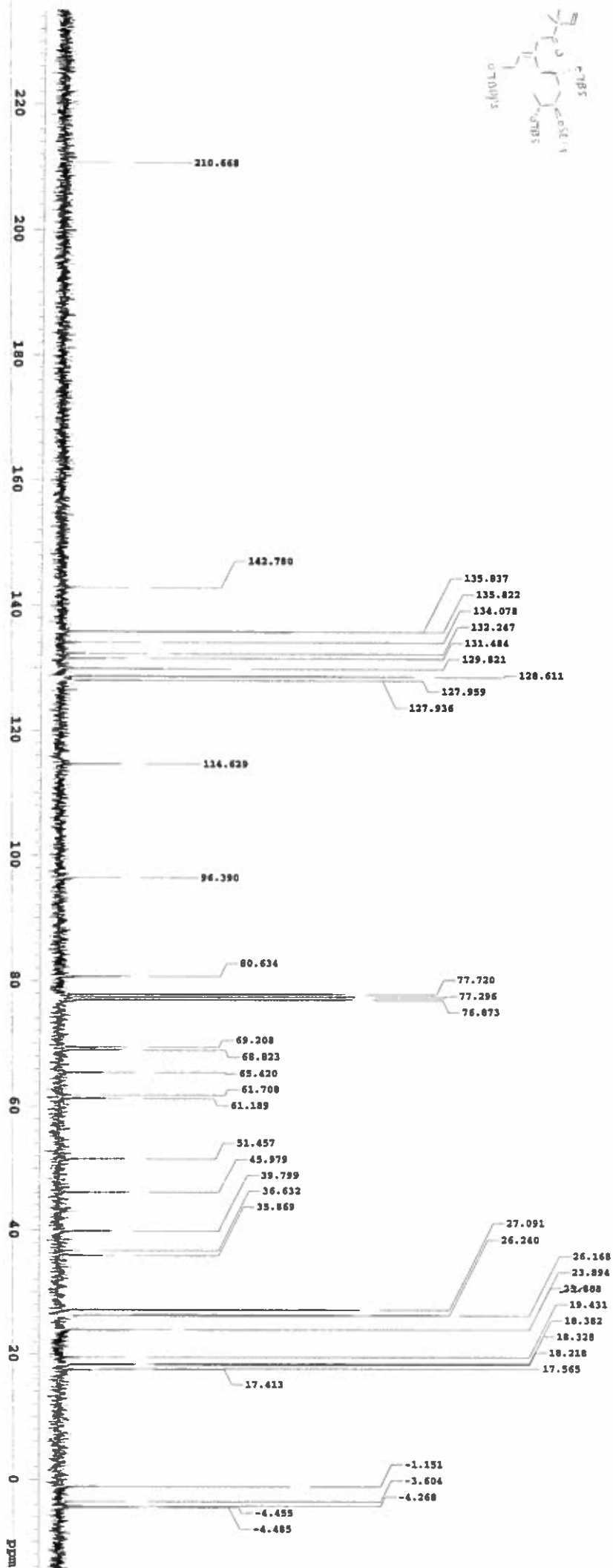
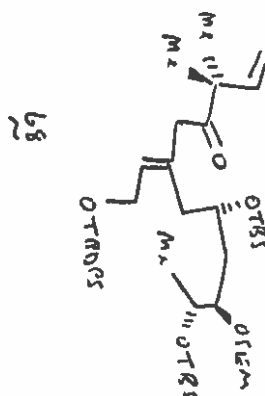
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec



867-3  
STANDARD IN OBSERVE

Automation directory: /data/metadata/Mar31  
File : 2601

Pulse Sequence: m2pul

Solvent:  $\text{cdcl}_3$ 

**Sample #26**

Wilco: 2601

**IMOVA-300 "ratty"**

Relax. delay 1.000 sec

Pulse 45.0 degrees

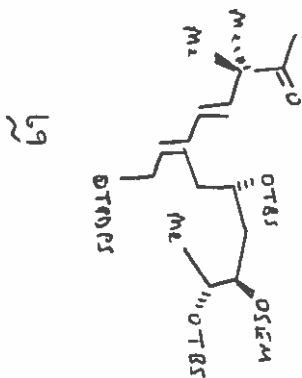
ACQ. TIME 3.744 sec  
Width 4797.6 Hz

16 repetitions

OBSEV# H1, 299.0458792 MHz  
DATA PROCESSING

RT #126 655336

**Total time 1 min, 16 sec**



c1g 383  
STANDARD 18 OBSERVE

Automation directory: /data/mmdata/mar24  
File: 1602

Pulse Sequence: zgpg30

Solvent: cdcl3

Ambient temperature

Sample 816

File: 1602

INSTR: 300 "recty"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 3.744 sec

Width 4797.6 Hz

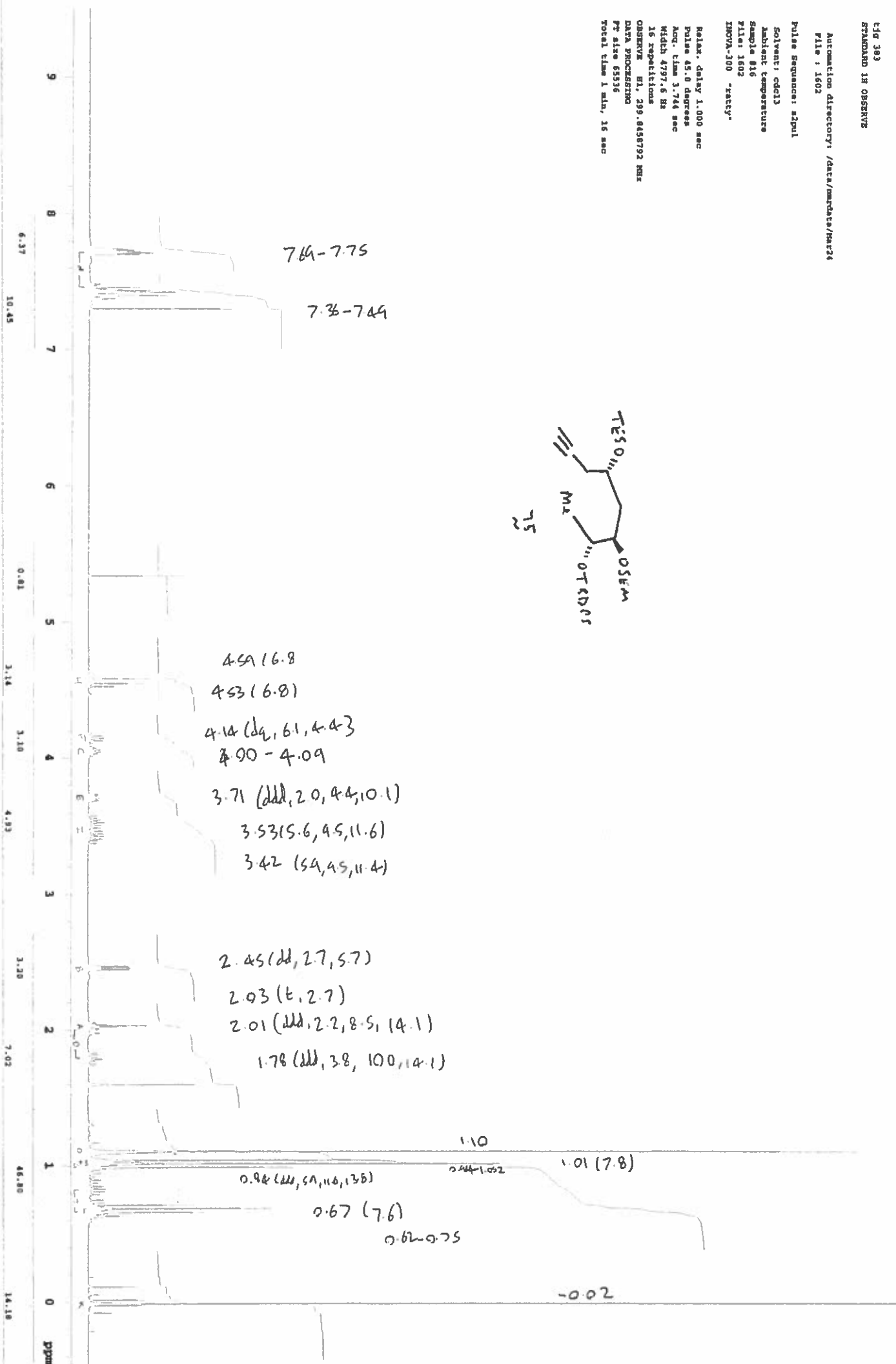
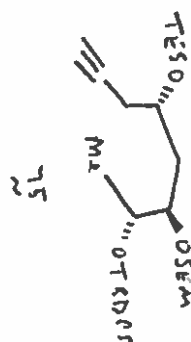
16 repetitions

OBSERVE: H1, 299.8458792 MHz

DATA PROCESSING

PT also 61516

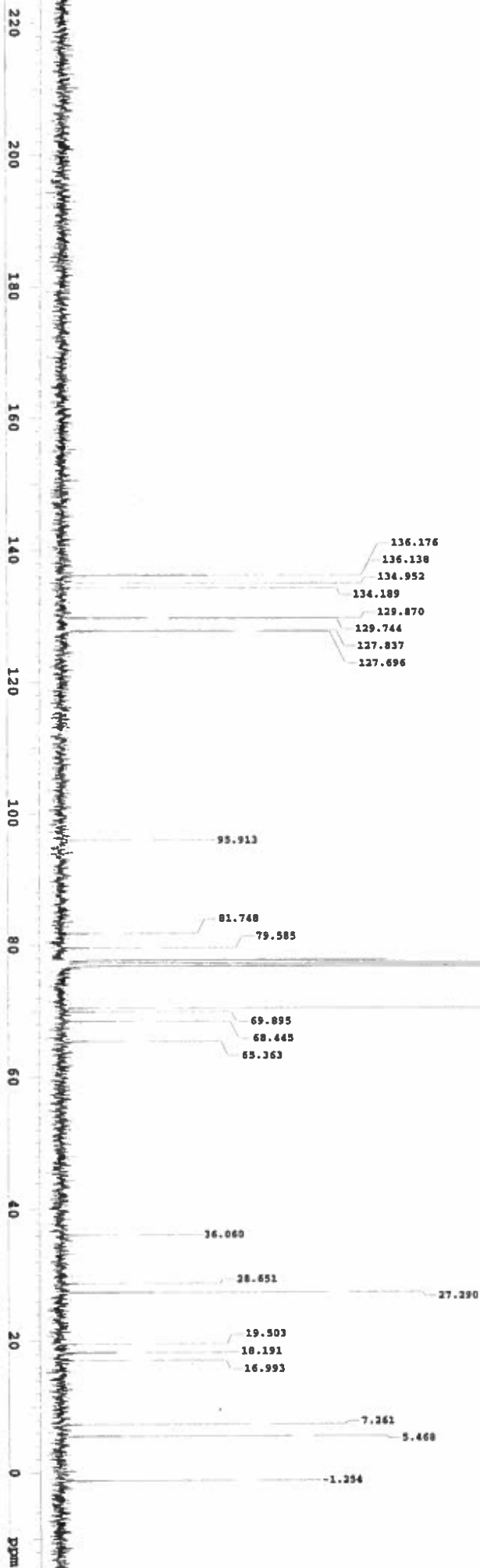
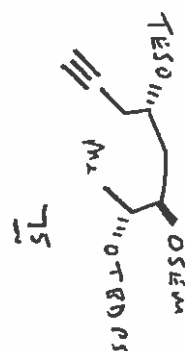
Total time 1 min, 16 sec



Automation directory: /data/mwdata/Haz24  
File : 1603

Pulse sequence: zgpg  
Solvent: cdcl3  
Ambient temperature  
Sample #16  
File: 1603  
INVTX-300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18850.1 Hz  
1024 repetitions  
OBSERVE C13, 75.3962883 MHz  
DECORPSE H1, 299.847786 MHz  
Power 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT also 131072  
Total time 48 min, 11 sec

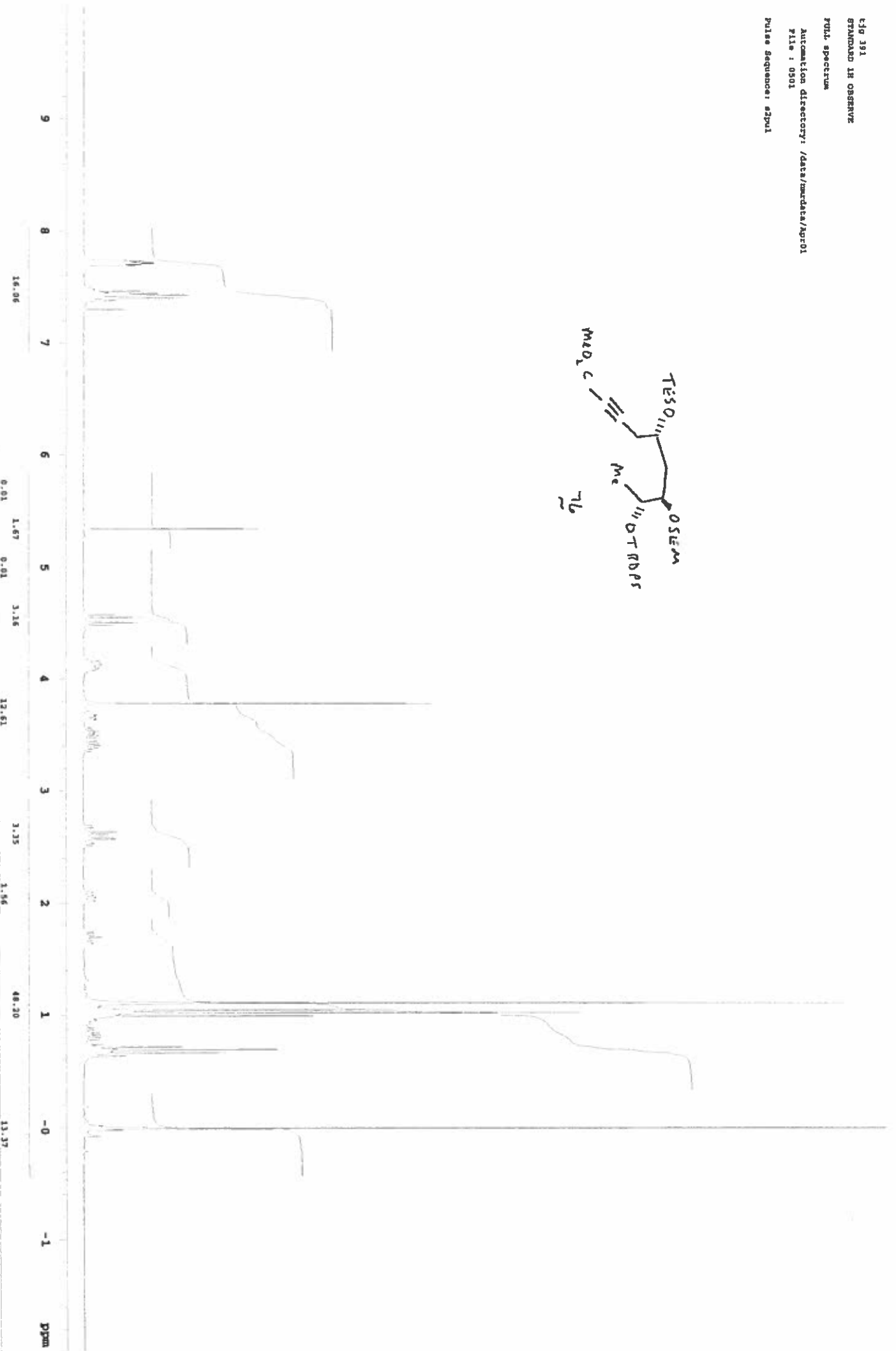
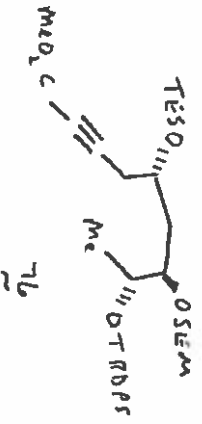


c3g 391  
STANDARD IN OBSERVE

FULL SPECTRUM

Automation directory: /data/mwdata/apr01  
File : 0501

Pulse sequence: zgpg1

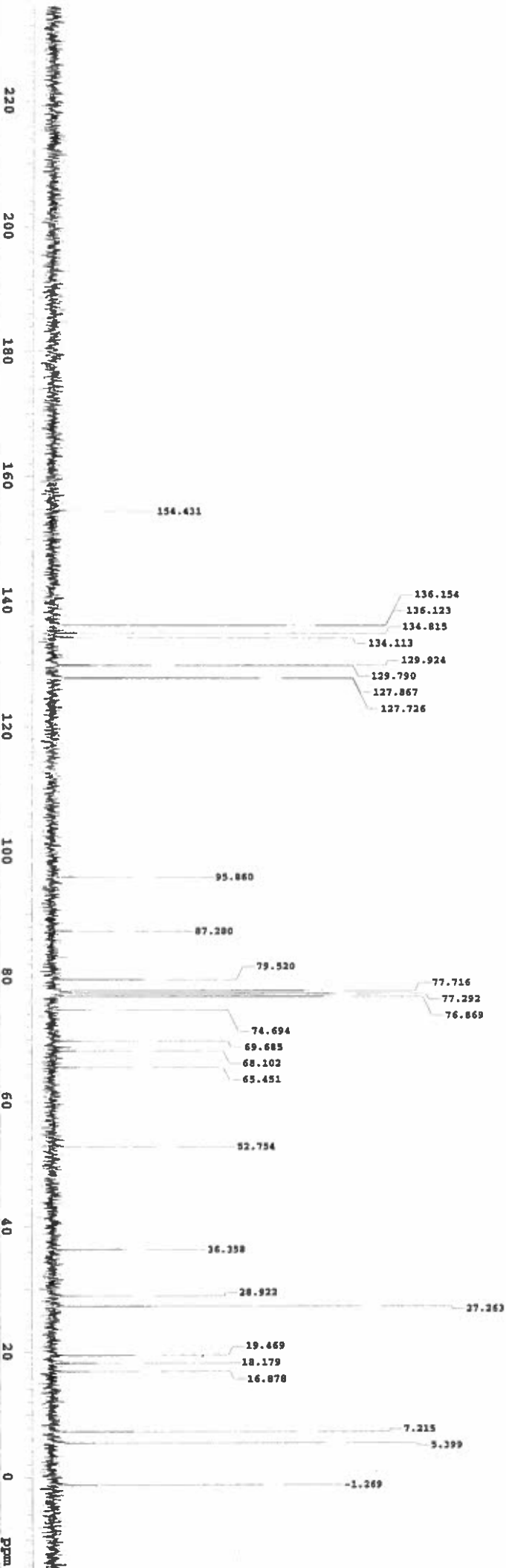
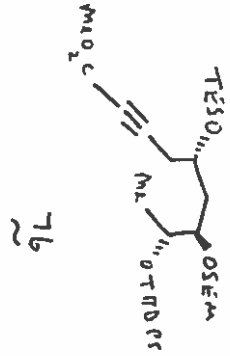


c3g 391  
STANDARD 1H OBSERVE

Automation directory: /data/mwdata/Apr01  
File: 0503

Pulse Sequence: w2pr1  
Solvent: cdcl3  
Ambient temperature  
Sample 85  
File: 0503  
INOVA-300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.015 sec  
Width 18850.1 Hz  
256 repetitions  
OBSERVE c13, 75.3962883 MHz  
DECOUPLE H1, 299.843786 MHz  
Power 37 dB  
Continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 11072  
Total time 12 min, 2 sec



EXP 530  
STANDARD 1H OBSERVE

Automation directory: /data/mrdata/32005  
File: 1201

Pulse sequence: zgpg30

Solvent: cdcl3

DATE Jan 6 2005

Sample 812

File: 1201

INSTRUMENT: spect

Pulse delay 1.000 sec

Pulse 45.0 degrees

Acq. time 3.766 sec

Width 4797.6 Hz

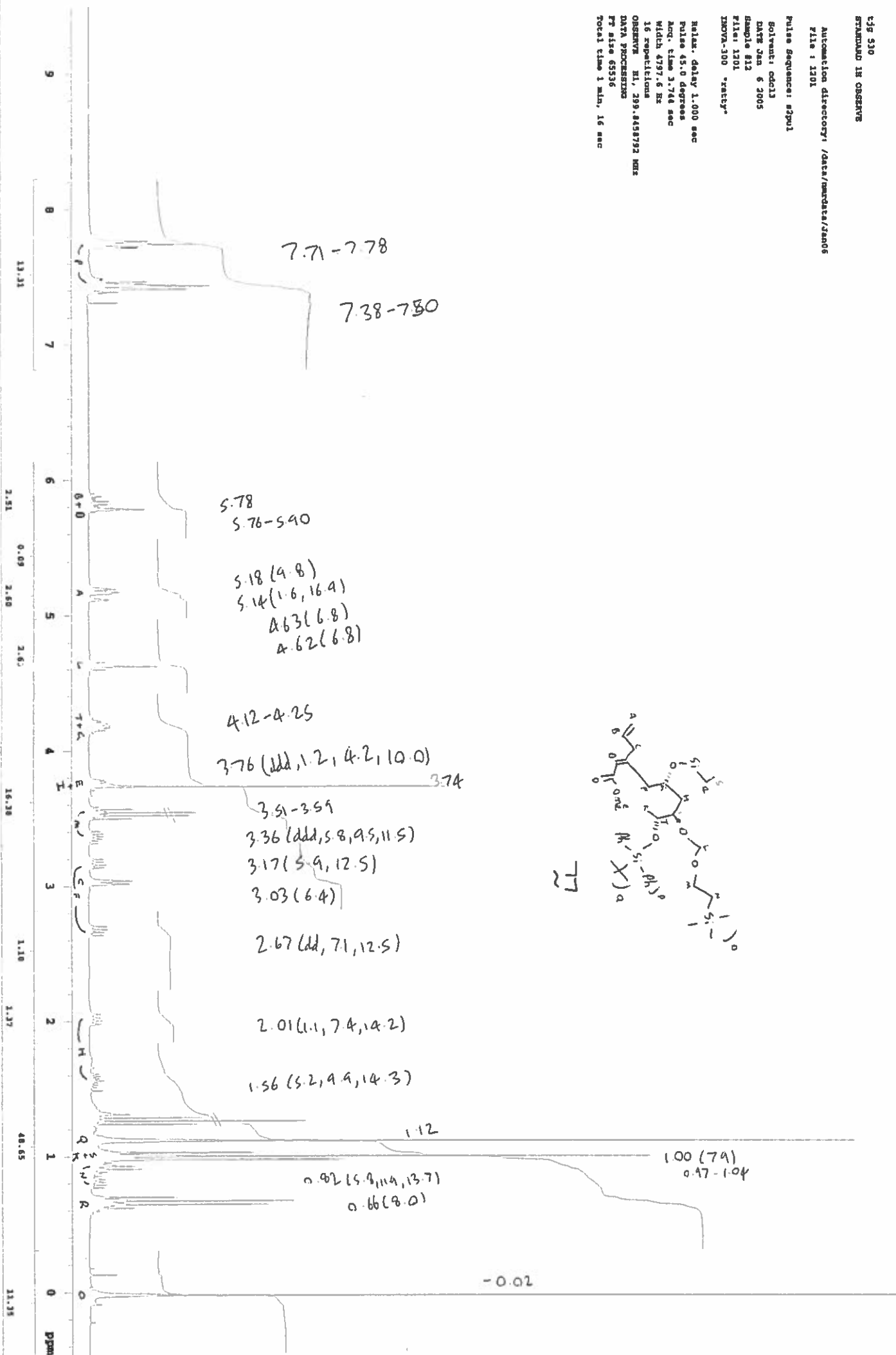
16 repetitions

OBSERVE: H1, 299.8458792 MHz

DATA PROCESSING

PF size 65536

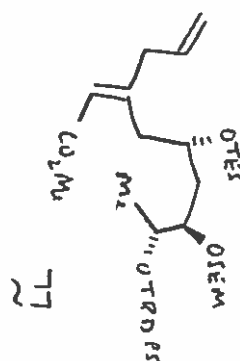
Total time 1 min, 16 sec



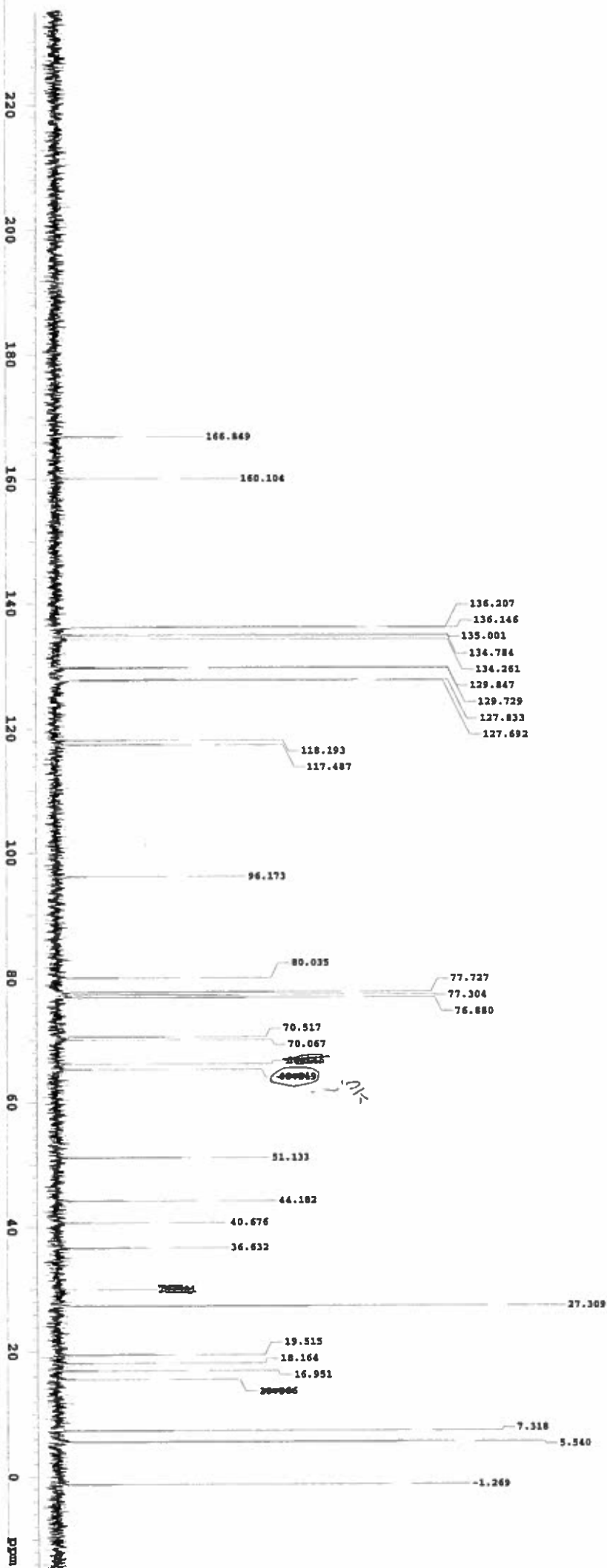
Automation directory: /data/monitor/2005  
File: 1202

Pulse Sequence: zgpg  
Solvent: cdcl3  
Date: Jan 6 2005  
Sample #12  
File: 1202  
INSTR: 300 "rally"

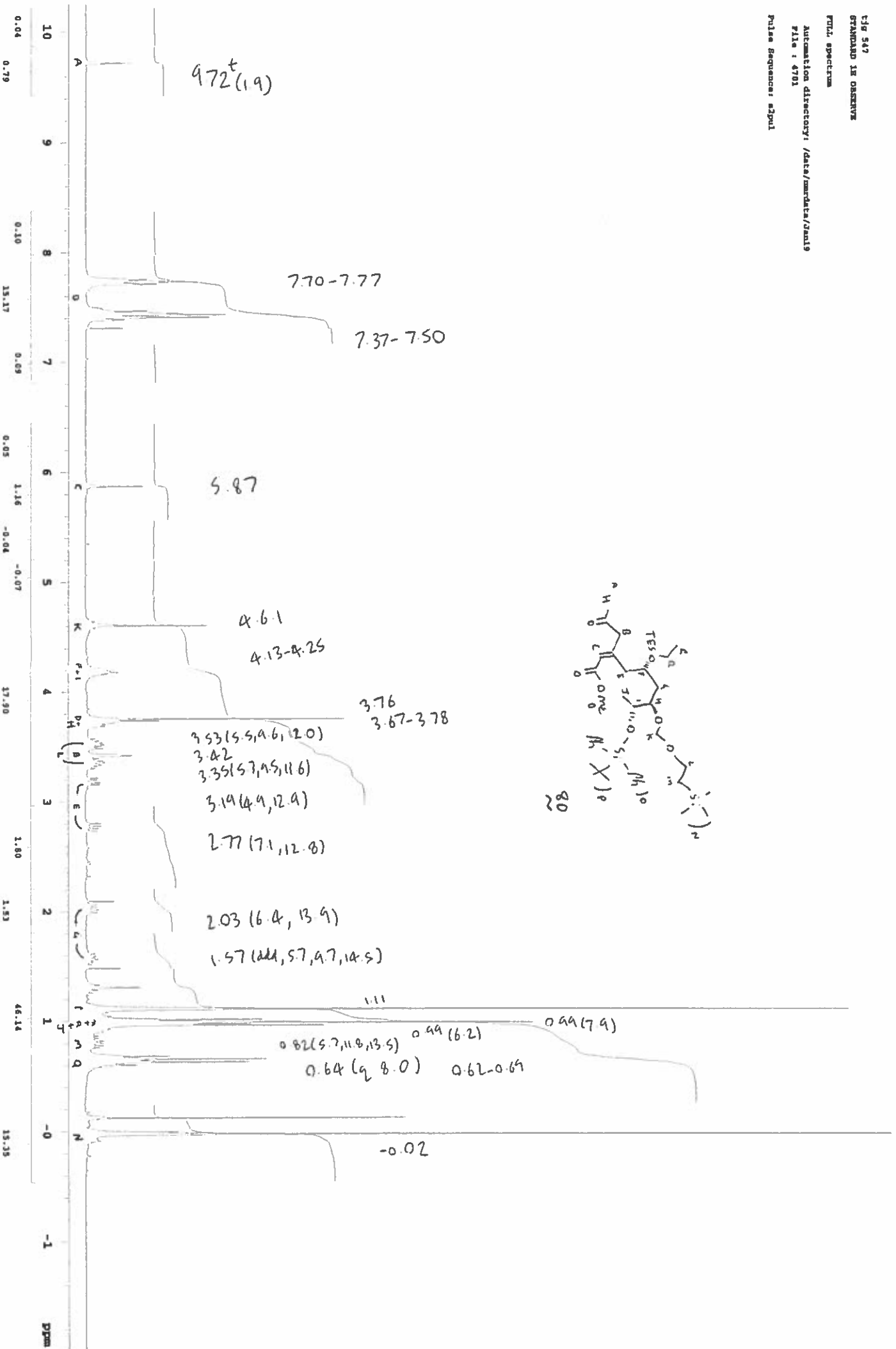
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18850.1 Hz  
356 repetitions  
OBSERVE CH: 75.3652883 MHz  
NUC1: 13C  
P1: 12.000 sec  
PC: 0.000 sec  
PT: 1.815 sec  
Total time 12 min, 2 sec



EtO acetate





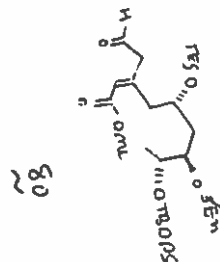
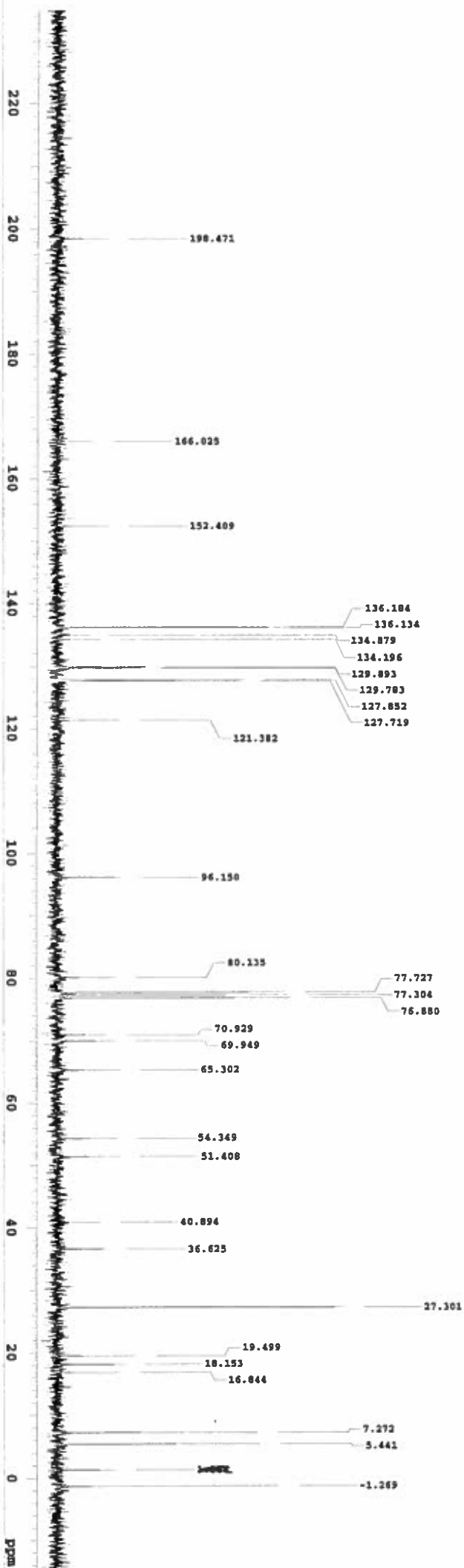


1:19 547  
STANDARD IN OBSERVE

Automation directory: /data/nmrdata/anal9  
File: 4703

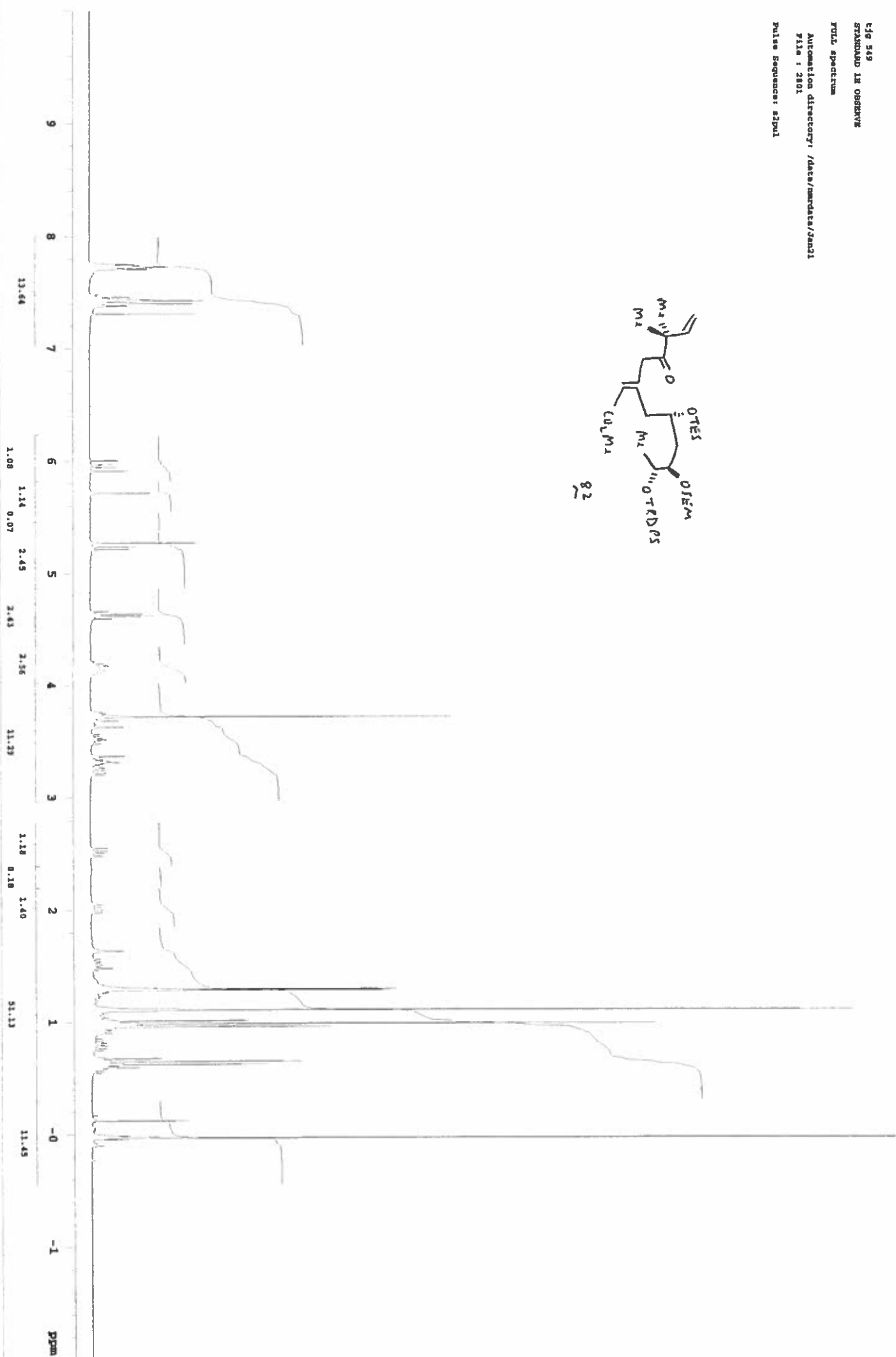
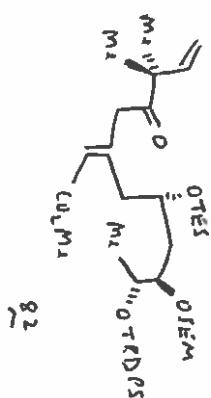
Pulse Sequence: zgpg30  
Solvent: cdcl3  
Date: Jan 19 2005  
Sample: 847  
File: 4703  
INOVA-300 "ratty"

Relax. delay: 1.000 sec  
Pulse: 45.0 degrees  
Acq. time: 1.015 sec  
Width: 16550.1 Hz  
256 repetitions  
OBSERVE: CL3, 75.362883 MHz  
DECOUPLE: H1, 299.8673786 MHz  
Power: 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening: 1.0 Hz  
FT size: 131072  
Total time: 12 min, 2 sec



### Total spectrum

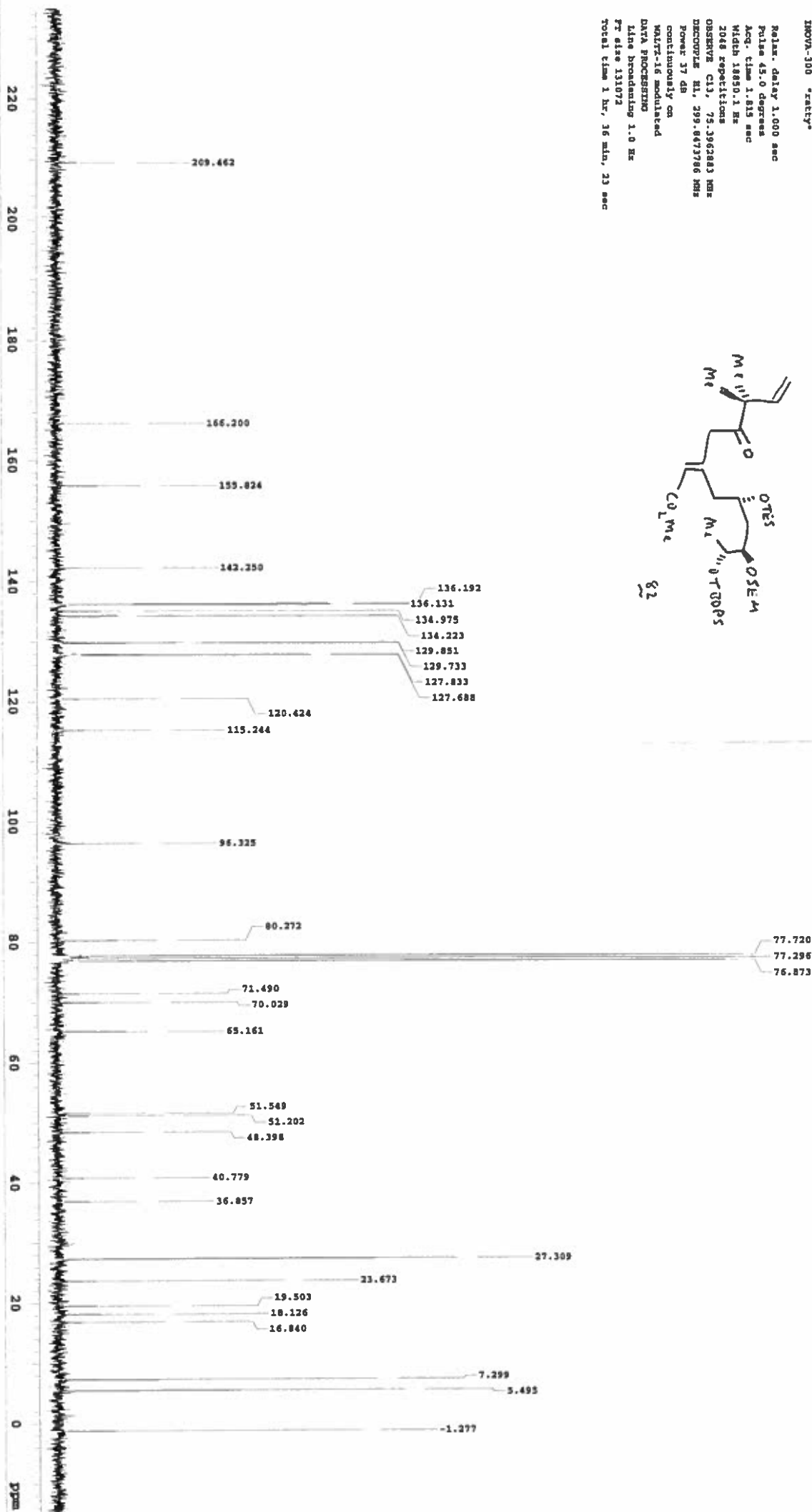
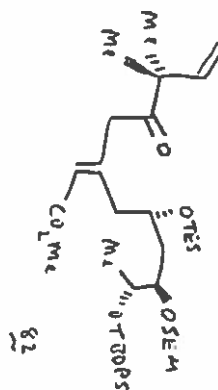
```
Automation directory: /data/tmrdate/3a021
File: 2802
Pulse Sequence: szpul
```



Automation directory: /sacs/nmrdata/2a021  
File: 0806

Pulse Sequence: zgpg1  
Solvent: cdcl3  
DATE Jan 21 2005  
Sample 88  
File: 0806  
INSTR: 300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.815 sec  
Width 18850.1 Hz  
2048 repetitions  
OBSERVE C13, 75.362883 MHz  
DECOUPLE H1, 299.8473786 MHz  
Power 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
F2 size 131072  
Total time 1 hr, 36 min, 23 sec



STANDARD  
EJG 554

STANDARD  
EJG 554

Automation directory: /data/wwrdata/Jan27  
File : 4302

Pulse Sequence: s2pul

Bolyvent: cdc13

DATE Jan 27 2005

File: 4302

ИЖОУА-300 "Емлет"

delay. delay 1.000 sec

Pulse 45.0 degrees

Width 3043.7 H:

16 repetitions

## DATA PROCESSING

FT 0120 32768

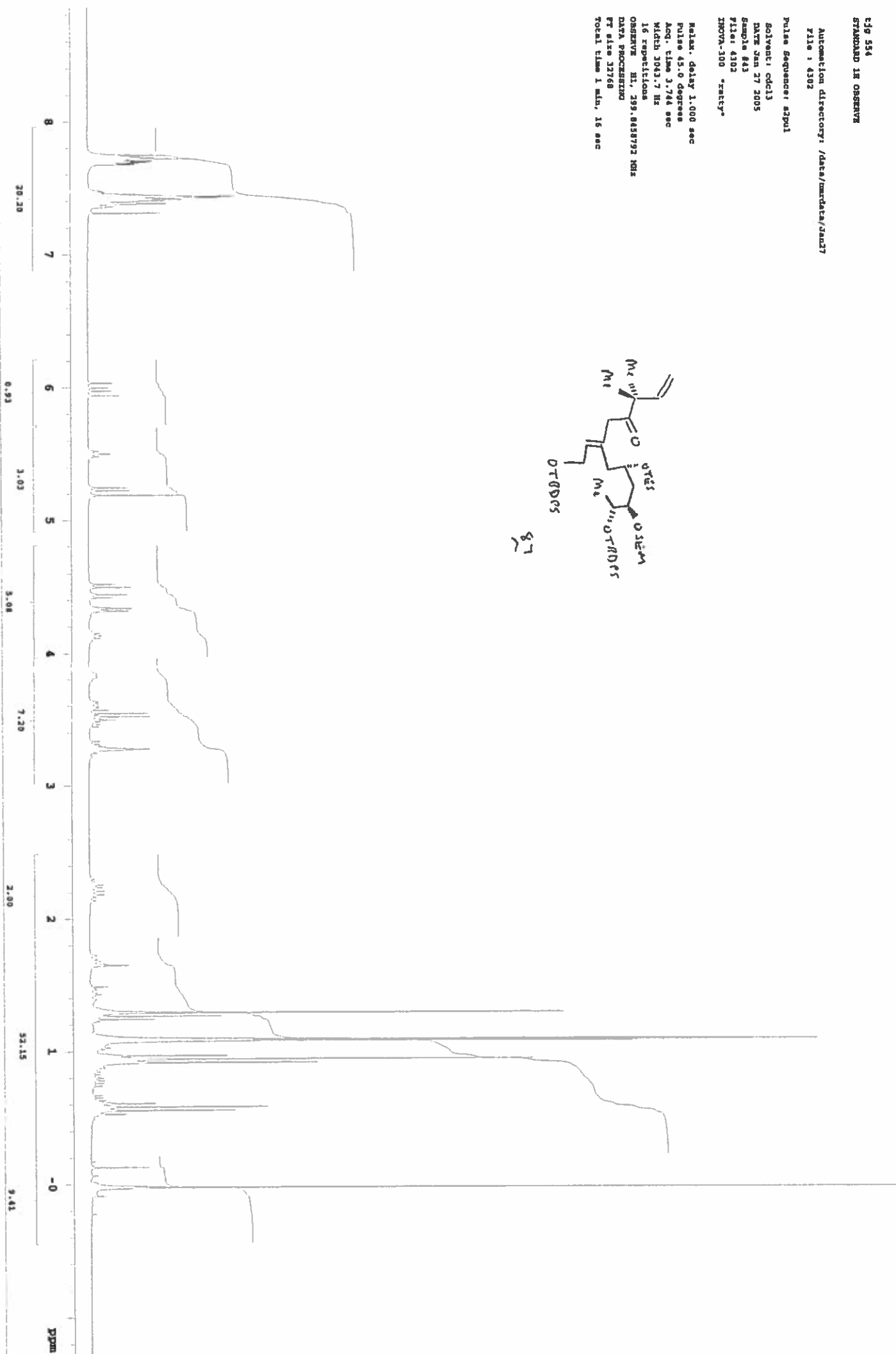
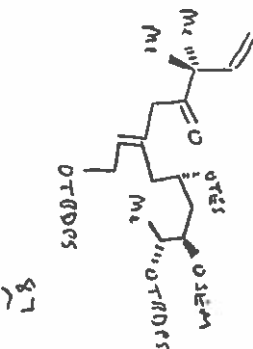


Fig 554  
STANDARD 1H OBSERVE

Automation directory: /data/rmdata/jan27  
File: 4303

Pulse sequence: zgpg1

Solvent: cdc13

DATE: Jan 27 2005

Sample 843

File: 4303

INSTR: 300 "ratty"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

256 repetitions

OBSERVE: C13, 75.1962883 MHz

DECOUPLE: H1, 299.8473786 MHz

Power 37 dB

continuously on

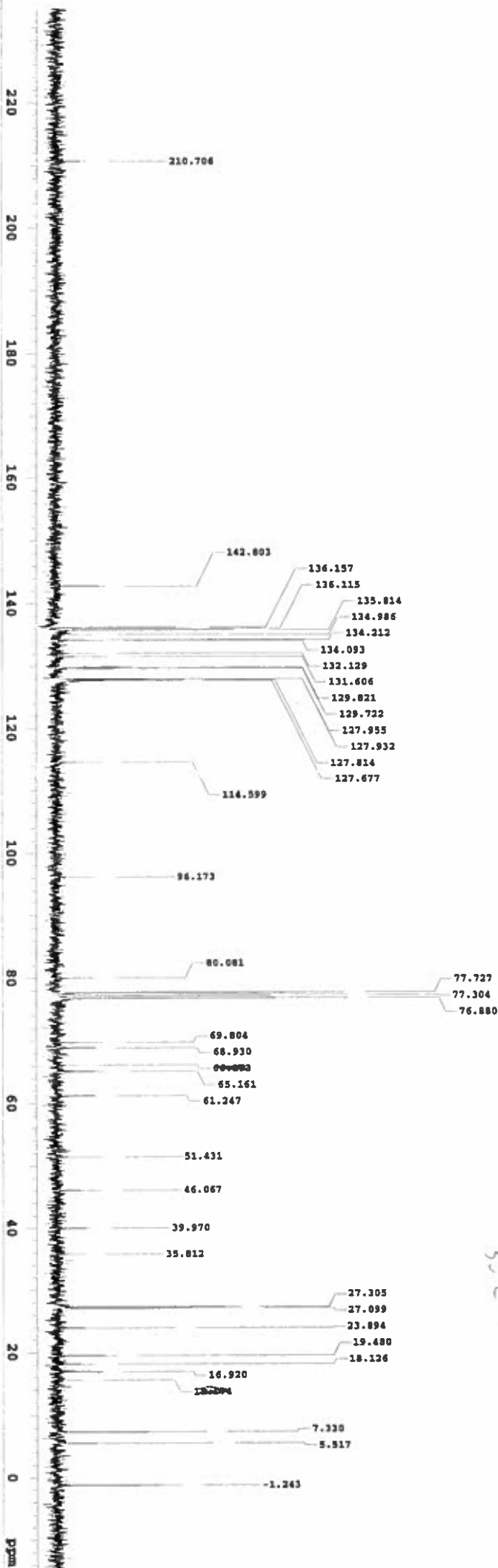
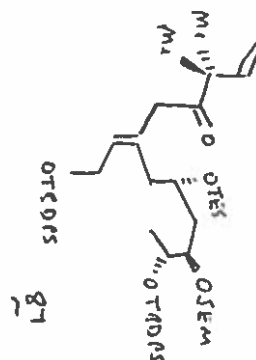
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec



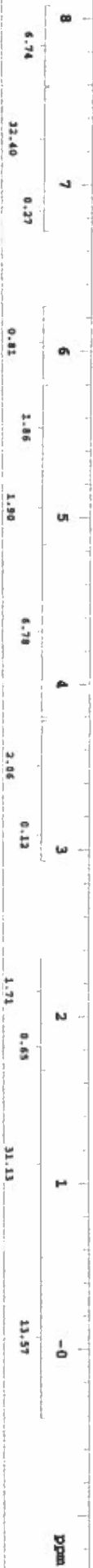
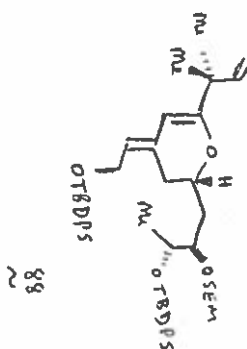
30.615

c3g 603  
STANDARD IN OBSERVE

Automation directory: /data/inmdata/Apr04  
File : 4902

Pulse Sequence: zgpg1  
Solvent: C6D6  
DATE Acq: 4/2005  
Sample 849  
File: 4902  
INOVA-300 "ratty"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 3.764 sec  
Width 3102.2 Hz  
16 repetitions  
OBSERVE H1, 299.8459062 MHz  
DATA PROCESSING  
F2 size 32768  
Total time 1 min, 16 sec



Automation directory: /data/nmrdata/apr04  
File: 4903

Pulse Sequence: zgpg30

Solvent: CDCl3  
Date Acq: 4/2005  
Sample: 849  
P1: 4.903  
PROVA-300 "ratty"

Relax. delay: 1.000 sec  
Pulse: 45.0 degrees  
Acq. time: 1.815 sec  
Width: 18850.1 Hz  
1024 repetitions  
OBSERVE: C13, 75.3962951 MHz  
NUC1: 13C, 299.8474056 MHz  
Power: 37 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening: 1.0 Hz  
F2 size: 131072  
Total time: 48 min, 11 sec

